BUTLER

May 20, 2015

Colleen E. Michuda, Esq.
United States Environmental Protection Agency
Atlanta Federal Center
61 Forsyth Street
Atlanta, GA 30303-8960

Re: Supplemental Information Request to Hercules, Inc.
EPA ID Number: MSD 008 182 081

Dear Colleen:

The purpose of this letter is to formally request an extension of time within which Hercules can respond to the Supplemental Information Request issued by EPA to Hercules dated April 21, 2015 (the "Request"). As you know from the several discussions in which you and I have engaged on this topic, Hercules has concerns about the scope of Request and relating to the ongoing civil litigation in which Hercules is currently involved. Although we have not provided any documents to you in response to the Request yet, Hercules is in the process of preparing documents responsive to this request and we appreciate your willingness to allow Hercules to respond to the Request in the stages we have discussed. As of today, I believe we have settled on a meeting date of June 3, 2015 for a meeting with you, Bill Weinischke of the United States Department of Justice, me, and Bill Reed of the Baker Donelson firm. I request that during that meeting, we discuss an appropriate timetable for providing documents responding to the Request. Hercules does not seek an indefinite extension for responding to this Request, but only an extension that will allow for a full discussion on June 3rd of an appropriate timetable for responding to the request as written.

Thank you for your consideration of this request and for your cooperation throughout this process.

Sincerely,

BUTLER SNOW LLP

John A. Brunini

cc: Kristina Woods

Post Office Box 6010
Ridgeland, MS 39158-6010

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June 9, 2015

VIA U.S. MAIL

Colleen E. Michuda, Esq.
United States Environmental Protection Agency
Region 4
Atlanta federal Center
61 Forsyth Street
Atlanta, Georgia 30303-8960

Re: Hercules Hattiesburg, Mississippi Facility - Poly-Pale and IB pH documents

Dear Colleen:

As we discussed in our call yesterday, enclosed please find a disc containing the Hercules poly-pale and impoundment basin pH documents.

If you have any questions about this information or any difficulty accessing it, please feel free to call or email me.

Sincerely,

BUTLER SNOW LLP

John A. Brunini

JAB:hat

Enclosures
26320924v1

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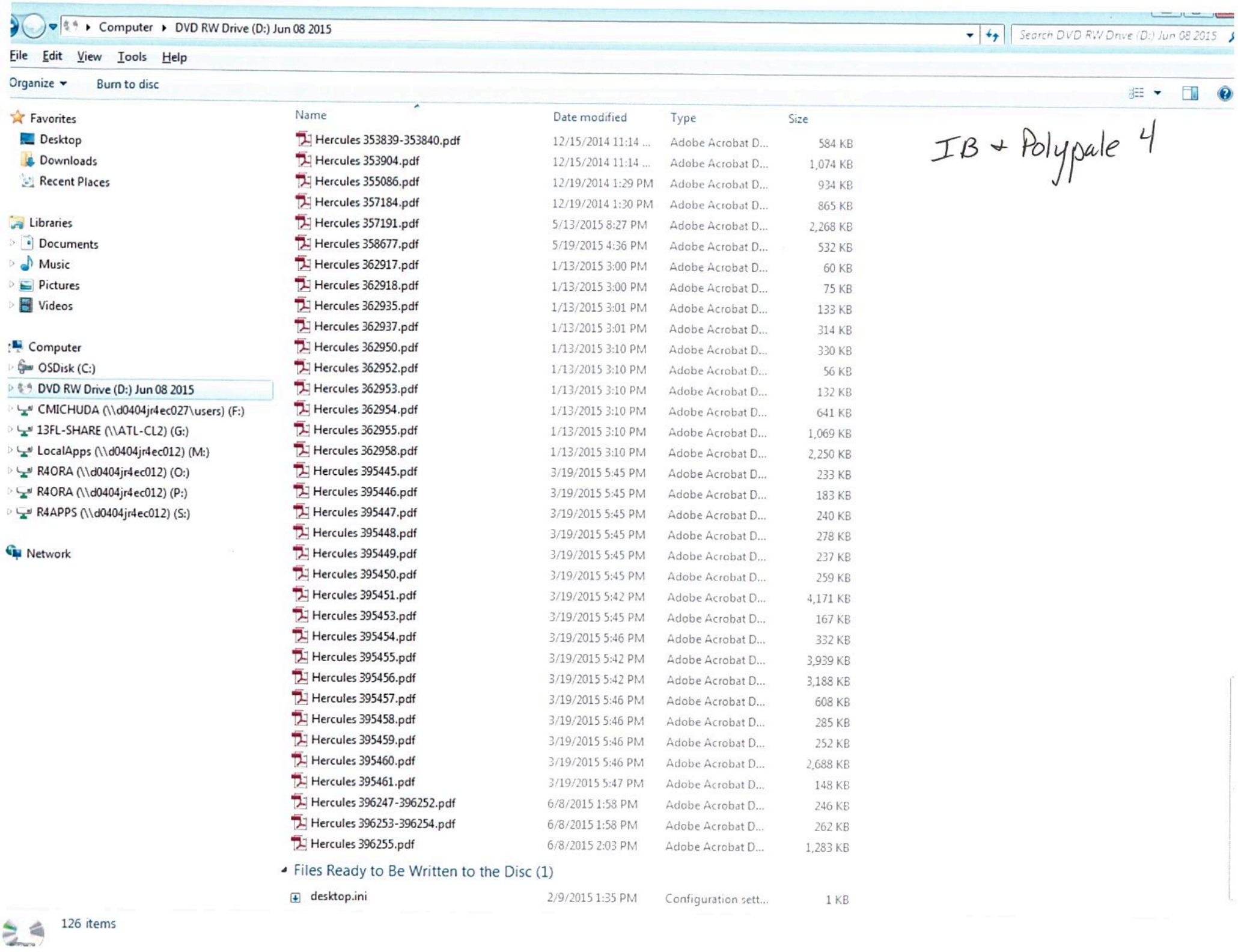

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June 25, 2015

VIA U.S. MAIL

Colleen E. Michuda, Esq.
United States Environmental Protection Agency
Region 4
Atlanta federal Center
61 Forsyth Street
Atlanta, Georgia 30303-8960

Re: Hercules Hattiesburg, Mississippi Facility - Poly-Pale and IB pH documents

Dear Colleen:

As you know, on June 9, 2015, on behalf of Hercules, I sent you a CD containing the Hercules poly-pale and impoundment basin pH documents we had agreed to provide. In the course of reviewing the last remaining project files prior to placement in storage, we have identified additional documents relevant to poly-pale and the impoundment basin pH issues. Enclosed with this correspondence, please find a disc containing these additional documents. If we discovery any additional documents relevant to these issues, we will provide copies to you. If you have any questions about this information or any difficulty accessing it, please feel free to call or email me.

Sincerely,

BUTLER SNOW LLP

John A. Brunini

JAB:hat
Enclosures

ButlerSnow 21292365v1
26622087v1

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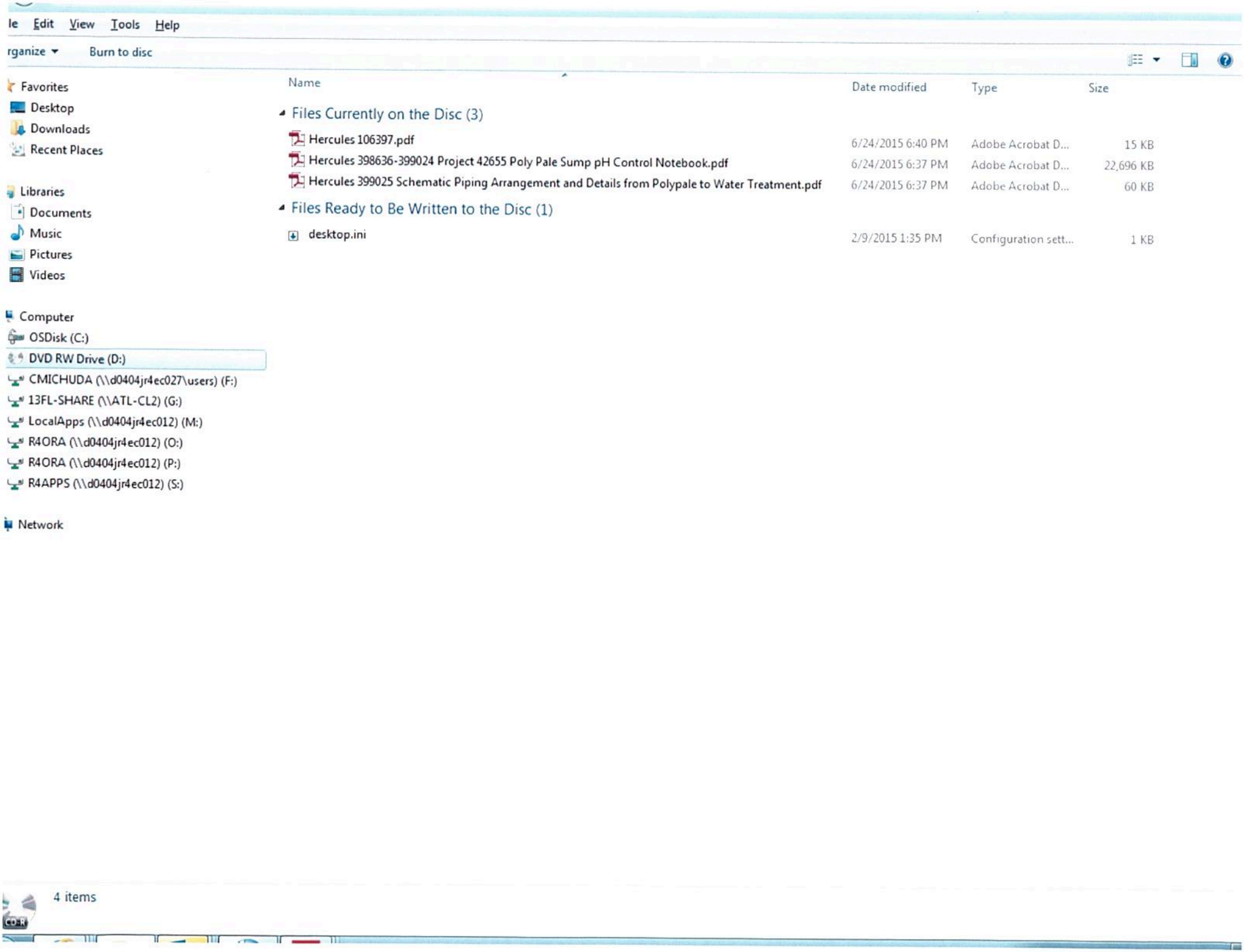
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November 10, 2015

Colleen E. Michuda, Esq.
Senior Attorney
Region 4
U.S. Environmental Protection Agency
61 Forsyth Street
Atlanta, GA 30303-8960

Re: Search of Relativity Database
Hercules Facility
Hattiesburg, Mississippi

Dear Colleen:

Enclosed please find a CD with documents containing the search terms you requested (Sludge /5 "drying beds" and Delnav /5 sludge). As stated in my email to you today, the number of documents on the enclosed CD will be different than the total number of hits in the database because duplicate and privileged materials have been removed. After reviewing this information, please feel free to call me with any questions.

Sincerely,

BUTLER SNOW LLP

John A. Brunini

cc:

20670459v 1

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Ridgeland, MS 39158-6010

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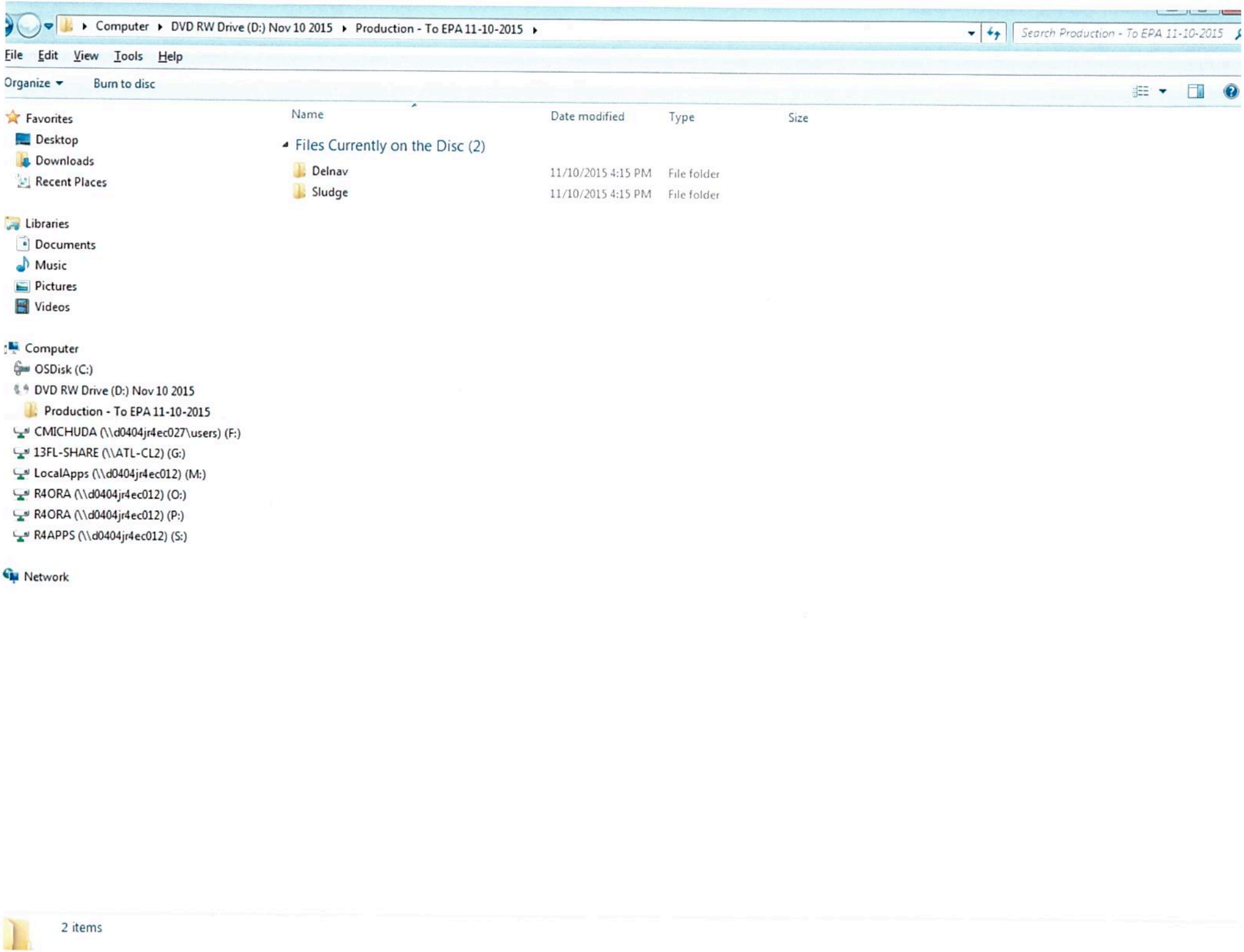
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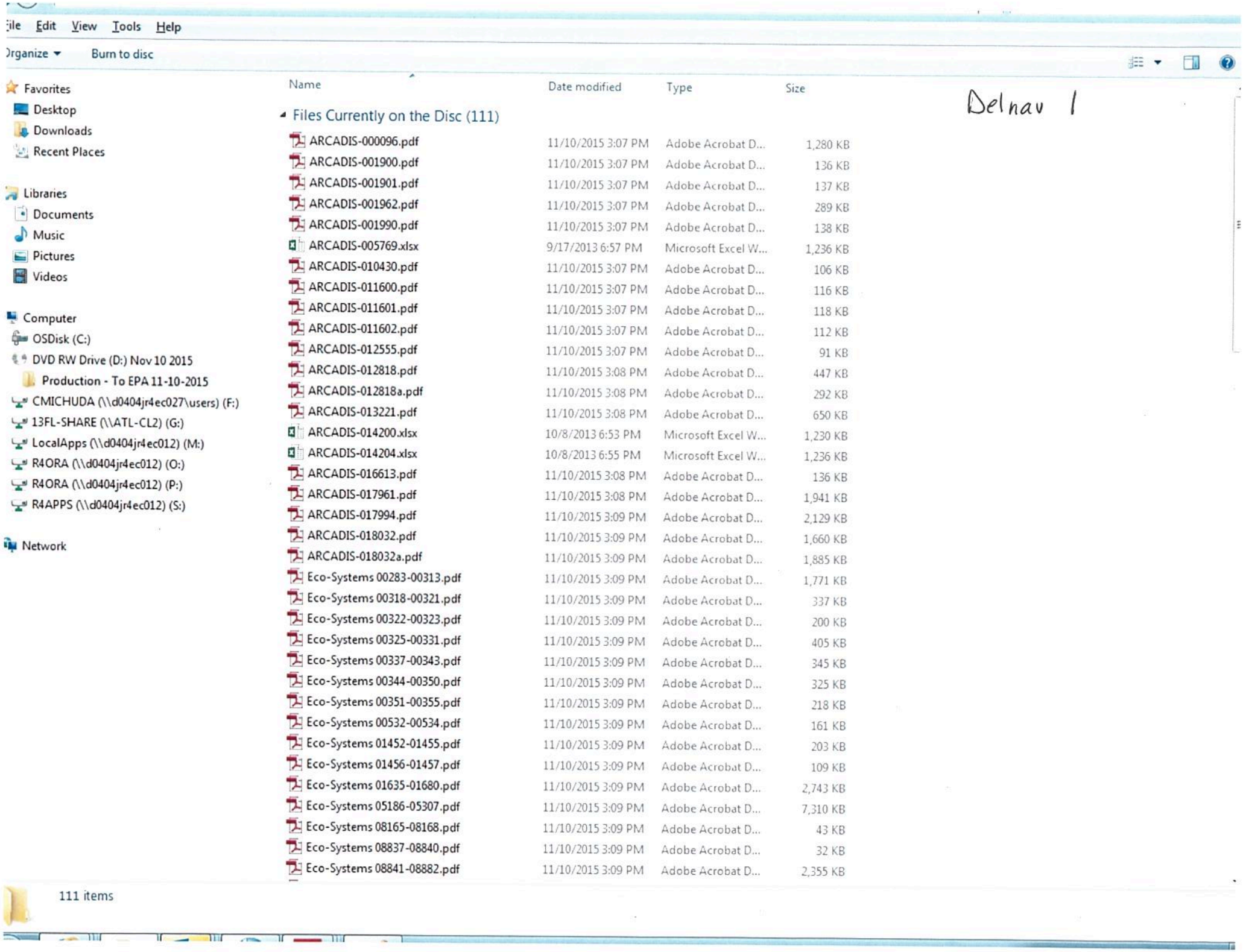

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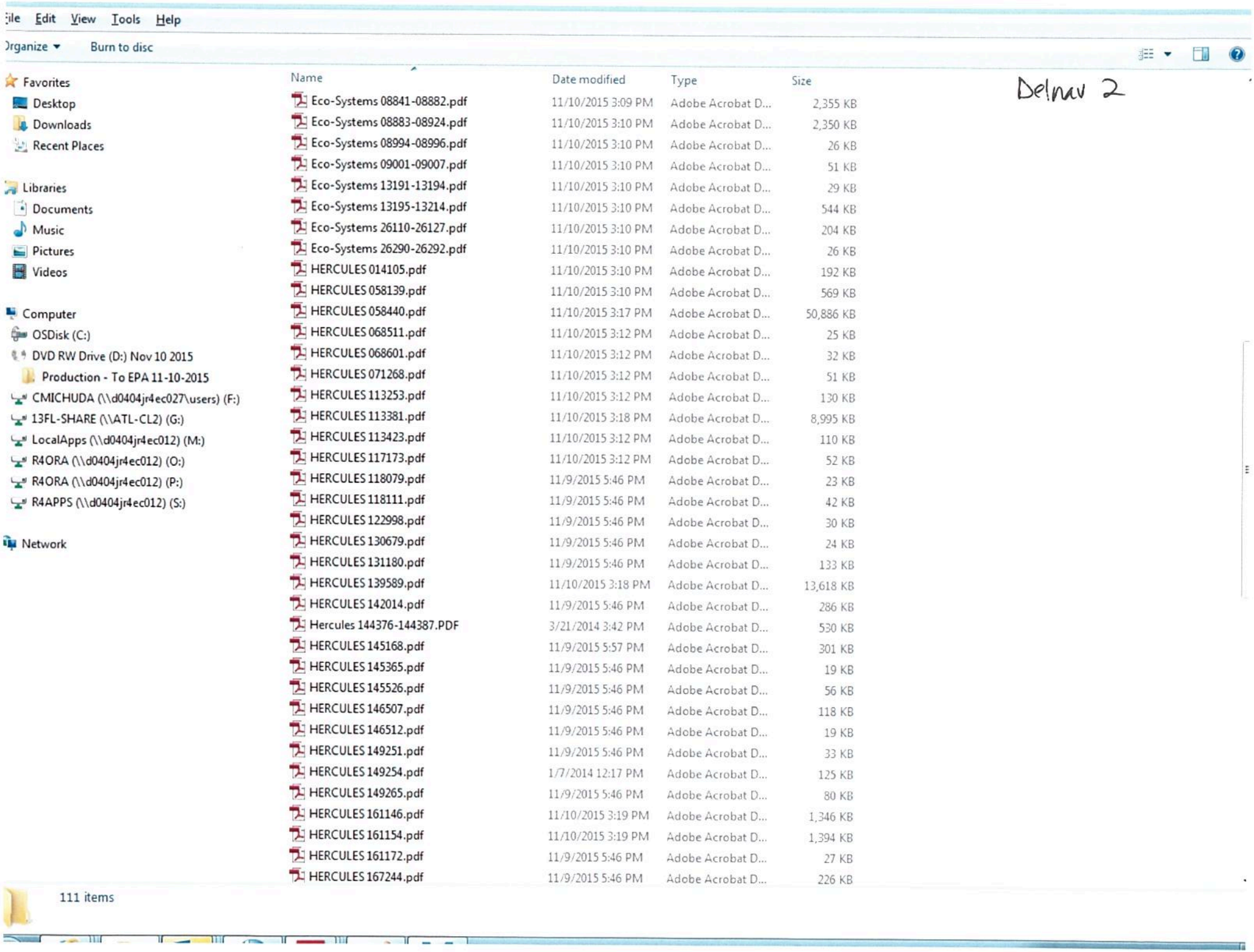
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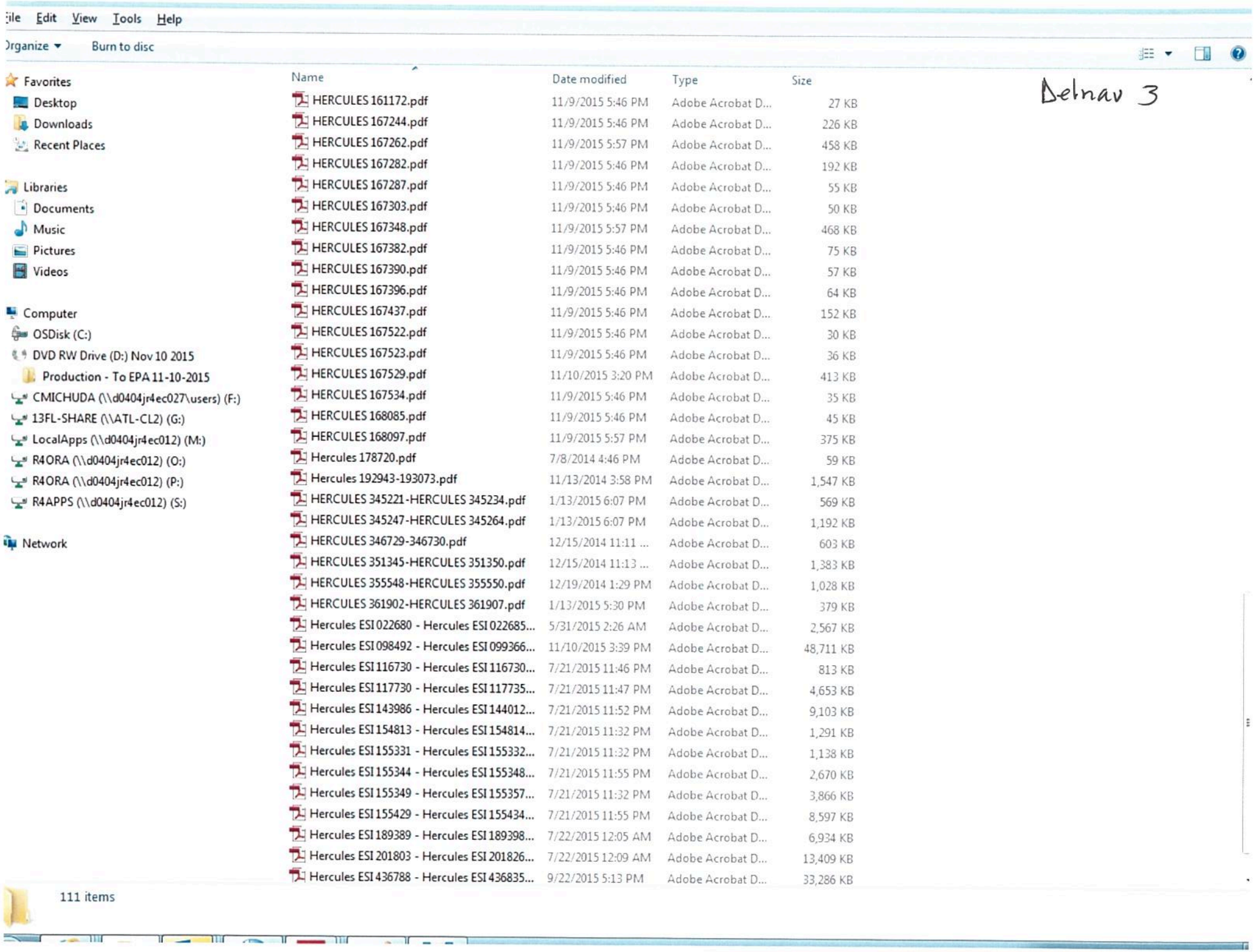
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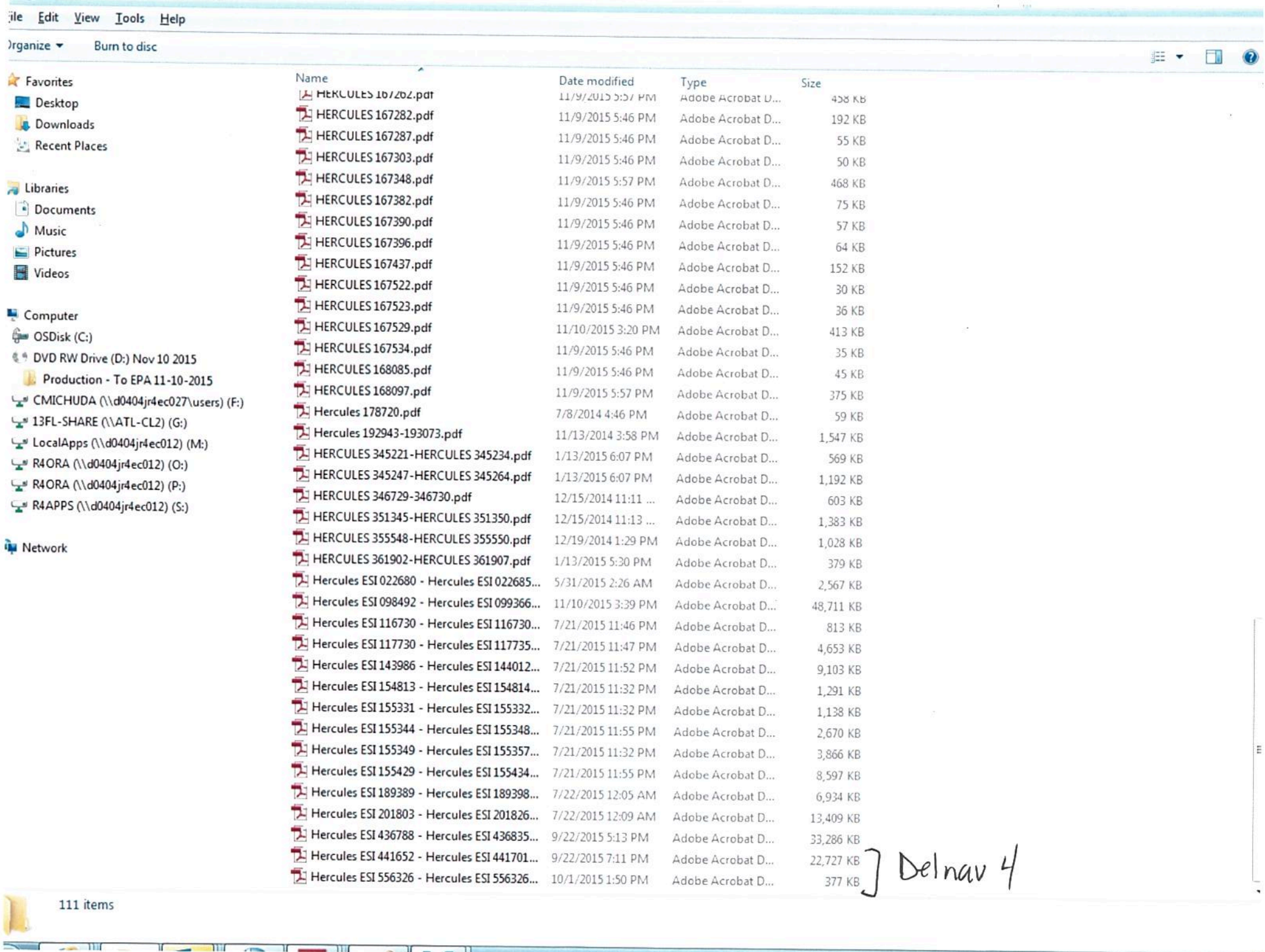
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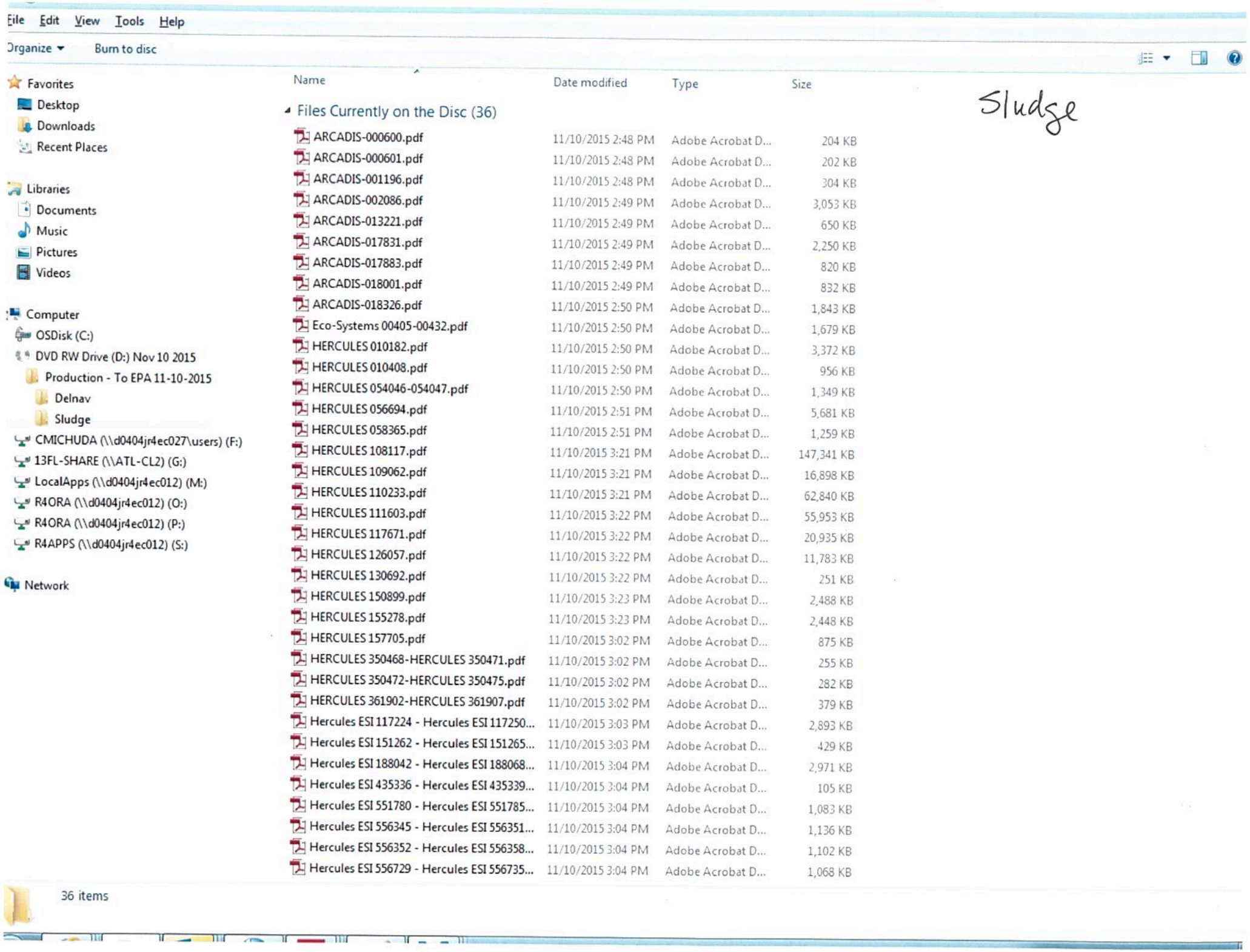
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Sludge

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4
ATLANTA FEDERAL CENTER
61 FORSYTH STREET
ATLANTA, GEORGIA 30303-8960

November 25, 2015

Via Electronic Mail and U.S. Mail

Mr. John Brunini
Butler Snow LLP
P.O. Box 6010
Ridgeland, Mississippi 39158-6010

Re: Application of K041 Listing to Sludge in the Hercules Impoundment Basin and Sludge Pits

Dear Mr. Brunini:

Thank you for your October 5, 2015 letter (hereinafter "Brunini letter") regarding Hercules Incorporated's (Hercules) analysis of whether the sludge in the Hattiesburg Impoundment Basin (IB) and Sludge Pits is properly classified as a K041 listed hazardous waste. Although the U.S. Environmental Protection Agency and the Mississippi Department of Environmental Quality appreciate Hercules' analysis, the agencies disagree with Hercules' conclusion that the K041 listing does not apply. As discussed in more detail below, Hercules' production of toxaphene at the Hattiesburg facility resulted in the generation of K041 listed waste. Pursuant to the mixture rule, the entirety of the sludge within (or excavated from) the IB is a K041 listed waste.[1] A detailed analysis of the regulatory status of the sludge at the Hattiesburg facility is set forth below.[2]

I. Characterization of the sludge in the IB as a K041 listed waste is governed by RCRA and mandated by the mixture and derived-from rules.

A proper analysis of the characterization of Hercules' toxaphene waste must begin with an overview of the Resource Conservation and Recovery Act (RCRA), 42 U.S.C. §§ 9601 *et seq.*, which serves as the foundation of hazardous waste management in the United States. Congress passed RCRA in 1976 as a comprehensive environmental statute providing the EPA and delegated states with authority to regulate hazardous waste from "cradle to grave." *City of Chicago v. Environmental Defense Fund*, 511 U.S. 328, 331 (1994). Pursuant to Section 3001 of RCRA, 42 U.S.C. § 6921, Congress tasked the EPA with promulgating regulations identifying characteristics of hazardous waste and listing particular hazardous wastes. Both characteristic and listed hazardous wastes are subject to the management standards of Subtitle C of RCRA, which includes regulations governing the generation, transportation, treatment, storage and disposal of hazardous wastes. *See* 42 U.S.C. §§ 6921-6925.

Over the years, the EPA has published several lists of specific "listed" hazardous wastes. *See* 40 C.F.R. Part 261, Subpart D, incorporated by reference at 11 Miss. Admin. Code Pt. 3, R. 1.2. Among the lists of hazardous waste are K-listed wastes, which include wastes from specific sources. *See* 40 C.F.R. § 261.32(a), incorporated by reference at 11 Miss. Admin. Code Pt. 3, R. 1.2. In general, "hazardous

[1] The focus of this letter is on characterization of the sludge within, or excavated from, the IB. Based on Hercules' historical removal campaigns within the IB, it is likely that K041 sludge was also disposed of in the Sludge Pits.
[2] The views expressed by the agencies in this letter are in response to the arguments set forth in Hercules' October 5th letter. To the extent these issues are raised in a separate forum or at a later date, the agencies remain free to supplement their positions and the supporting information underlying them, as the agencies deem appropriate.

waste listings are retroactive, so that once a particular waste is listed, all wastes meeting that description are hazardous wastes no matter when disposed." *Chemical Waste Mgmt., Inc. v. EPA,* 976 F.2d 1526, 1530-31 (D.C. Cir. 1989). Thus, "[i]f an abandoned site is being remediated and wastes or contaminated media are being removed from the site, any wastes meeting the listing descriptions . . . must be managed in accordance with all applicable requirements." 57 Fed. Reg. 37,284, 37,298 (Aug. 18, 1992).

Further, under the mixture and derived-from rules, any solid wastes that are either mixed with a listed waste or generated from the treatment, storage, or disposal of a listed waste, are also considered to be listed wastes. *See* 40 C.F.R. §§ 261.3(a)(2)(iv) and 261.3(c)(2)(i), adopted by reference at 11 Miss. Admin. Code Pt. 3, R. 1.2. As early as 1980, the EPA recognized that this concept was a fundamental principle of hazardous waste identification and subsequent waste management. To find otherwise would allow generators to avoid Subtitle C regulation "simply by commingling listed wastes with nonhazardous solid waste." *See* 45 Fed. Reg. 33,084, 33,095 (May 19, 1980). As the court stated in *Chemical Waste Mgmt.*, 869 F. 2d at 1539, "a hazardous waste does not lose its hazardous character simply because it changes form or is combined with other substances." In fact, "it is reasonable to assume that all such mixtures and derivatives are hazardous until shown otherwise." *American Chemistry Council v. EPA*, 337 F. 3d 1060, 1065 (D.C. Cir. 2003). Ultimately, in upholding the mixture and derived-from rules, the court recognized that "Congress wanted the EPA, in deciding which substances to regulate as 'hazardous' under the RCRA, to err on the side of caution." *Id.* at 1066. As a result, the court concluded that the rules fulfill "the purpose for which Congress passed the RCRA, namely to subject hazardous waste to 'cradle-to-grave' regulation in order to protect public health and the environment." *Id.* at 1065.

Rather than erring on the side of caution, Hercules urges the agencies to adopt an extremely narrow interpretation of the scope of the K041 listing, in essence, allowing Hercules to do exactly what the mixture rule was designed to prevent – avoid regulation simply by mixing a listed waste with a solid waste and then asserting that the mixed waste no longer meets the listing description. The agencies simply cannot adopt such an interpretation.[3] There is no dispute that Hercules produced toxaphene from at least 1951 to 1952, that wastewaters from such production were treated in the IB, and that toxaphene has been detected in the IB in recent sampling events. The resultant sludge therefore meets the listing description for K041. As discussed above, hazardous waste listings apply retroactively, so the K041 listing applies despite the historic nature of the toxaphene production. Finally, given the manner in which the IB was managed when in use, application of the mixture rule results in the entirety of the IB sludge carrying a K041 listing. The EPA and MDEQ believe the discussion of whether the sludge at the facility is a K041 listed waste should end here. Unfortunately, Hercules has attempted to confuse the issues by questioning the source of the toxaphene, the wording of the listing, and the applicability of the mixture rule. Although none of these arguments has merit, the agencies will address each argument in turn below.

II. Hercules' production of toxaphene at the Hattiesburg facility is a source of the toxaphene in the IB.

In disputing the applicability of the K041 listing to the sludge at the Hattiesburg facility, Hercules first argues that the source of the toxaphene contamination in the IB and Sludge Pits is unknown. Hercules cites the EPA's October 14, 1998 Memorandum from Timothy Fields, Jr. and Steven A. Herman to the RCRA/CERCLA Senior Policy Managers and Regional Counsels entitled "Management of Remediation Wastes" (hereinafter, "Remediation Waste Memo"), which states that if documentation regarding the

[3] Comprehensive environmental statutes enacted to protect public health and the environment must be construed broadly so as to effectuate the goals of the statute. *See United States v. Sellers*, 926 F. 2d 410, 416 n. 2 (5th Cir. 1991) (finding that RCRA is entitled to liberal construction to effectuate its purpose of protecting public health).

source of contamination is "unavailable or inconclusive," one may assume that a waste is not a listed hazardous waste. *See* Brunini letter at p. 2. Hercules attempts to fit the Hattiesburg situation into the "unavailable or inconclusive" category under the guidance by hypothesizing about a number of other possible sources for the toxaphene found in the IB. Without producing any evidence of these other sources, Hercules downplays and dismisses the one known and undisputed source of toxaphene in the IB for which there *is* documentary evidence – the actual production of toxaphene at the Hercules Hattiesburg facility from at least 1951-1952. Hercules' own records reveal, and Hercules does not dispute,[4] that over 27 million pounds of toxaphene were produced at the Hattiesburg facility from 1951 to 1952. *See* Enclosure 1. In addition, it is undisputed that wastewaters from this production process went to the IB for treatment. *See* Enclosure 2 (stating that the toxaphene wastewater passes through "an impounding (sedimentation) basin, where it is combined with and diluted by other plant waste water").

Given this available documentary evidence, the Remediation Waste Memo upon which Hercules relies actually contradicts Hercules' conclusion that the sludge within the IB is not a K041 listed waste. The Remediation Waste Memo specifically recommends that lead agencies "use available site information such as manifests, storage records, and vouchers in an effort to ascertain the sources of wastes or contaminants." Remediation Waste Memo at p. 5. This situation, unlike many Superfund sites, involves numerous, available records regarding historical production processes or operations at the facility. As a result, it is disingenuous for Hercules to now argue that the source of the toxaphene in the IB is "unknown" by hypothesizing about a plethora of undocumented and speculative sources of toxaphene and to then argue that those speculative and undocumented sources (rather than the documented production of 27 million pounds of toxaphene) are more likely to be the source of the toxaphene in the IB.[5] In addition, even if there were other potential sources of the toxaphene contamination in the IB, the presence of another contributing source would not negate the fact that the production of toxaphene (specifically covered by the K041 listing description) is *one* of the sources, and under the mixture rule (discussed above), all of the resultant sludge is a K041 listed waste.

III. The K041 listing covers toxaphene sludges produced at the Hattiesburg facility.

Hercules next contends that it "never produced a sludge within the scope of the K041 listing," injecting both a requirement of purity and a threshold concentration limit into the text of the K041 listing. *See* Brunini letter at p. 4. Specifically, Hercules argues that the K041 listing is "limited to highly concentrated sludges from wastewater treatment systems dedicated to managing toxaphene production wastewaters." *Id.* at p. 2. Hercules claims that the listing was not intended to apply to "less concentrated sludges from non-dedicated systems." *Id.* Hercules also asserts that concentrations of 10,000 milligrams/kilogram (mg/kg) or more of toxaphene are necessary in order for the sludges to fall within the listing description.

As a threshold matter, several toxaphene wastes are listed hazardous wastes: (1) wastewater treatment sludge from the production of toxaphene (K041); (2) untreated process wastewater from the production of toxaphene (K098); and (3) discarded commercial chemical products, off-specification species, container residues, and spill residues thereof (P123). *See* 40 C.F.R. §§ 261.32 and 261.33, adopted by

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[4] Hercules' July 28, 2011 Information Request Response to the EPA confirms that Hercules "manufactured, produced, and managed" toxaphene at the Hattiesburg facility. *See* Hercules Information Request Response, A. 10.

[5] Hercules raises a number of other potential sources of the toxaphene in the IB on pages 3-4 of the Brunini letter; however, given the hypothetical nature of these sources and the lack of documentation upon which the EPA or MDEQ could rely, the agencies will not address the arguments presented, including the arguments that the wastewater mixtures are exempt under 40 C.F.R. §§ 261.3(a)(2)(iv) or 261.3(c)(2)(i). Although the agencies acknowledge that toxaphene may have been used in the State of Mississippi during the 1970s (along with several other states, as Hercules points out, "from Texas to Georgia"), the EPA and MDEQ are not persuaded that the toxaphene in the IB and the Sludge Pits is from the use of toxaphene generally in the southeastern United States.

reference at 11 Miss. Admin. Code Pt. 3, R. 1.2. The EPA has found that toxaphene "is an exceptionally dangerous waste constituent" and that it is extremely toxic, highly bioaccumulative, mutagenic, and reported to cause cancer in laboratory animals. *See* Background Listing Document (November 14, 1980) at p. 7. At the time of the 1980 listing of K041 and K098, toxaphene was found to bioaccumulate in environmental receptors by factors as much as 300,000. *See id.* at p. 9. By 1990, the United States had cancelled all toxaphene use in the United States. *See* http://www3.epa.gov/airtoxics/hlthef/toxaphen.html. As a result, Hercules' narrow and erroneous interpretation of the K041 listing is completely at odds with the purpose and plain language of the listing, and would undermine the structure of RCRA and its associated hazardous waste management regulations.

A. The K041 listing is not limited to the Hercules Brunswick facility or the Vertac Chemical facility.

As a threshold matter, Hercules first attempts to argue that only the facilities and processes specifically referenced in the K041 listing document (the Hercules Brunswick, Georgia facility and the Vertac Chemical Vicksburg, Mississippi facility) are covered by the K041 listing. *See* Brunini letter at p. 5. This interpretation would require the EPA to enumerate specific facilities in every rulemaking in order for the rulemaking to apply. The K-listed wastes in 40 C.F.R. § 261.32, adopted by reference at 11 Miss. Admin. Code Pt. 3, R. 1.2, are based on "specific sources," not specific individual facilities. In addition, there is nothing in the text of the listing or in the Background Listing Document that suggests such a limitation on the applicability of the listing. Under Hercules' argument, any facilities that begin to generate wastes meeting a listing description after such listing has been promulgated could never be covered by the listing as they could not have been referenced in the relevant background listing document. This proposition is completely at odds with RCRA's regulatory framework regarding the identification and listing of hazardous wastes. In fact, the Agency specifically rejected the concept that wastes should be listed on a "site-by-site" basis, determining that it was well within Congress' intent that EPA list wastes generically by class. *See* 45 Fed. Reg. 33,084, 33,114 (May 19, 1980) (rejecting the proposition that wastes could only be listed on a "generator by generator" basis and not by class).

B. The K041 listing is not limited to pure toxaphene waste streams.

Second, Hercules argues that because the K041 Background Listing Document cites the Hercules Brunswick facility as an example of a facility that produced toxaphene at the time of the 1980 listing, and because the Brunswick facility used a dedicated wastewater treatment system to treat its toxaphene wastewaters, that the K041 listing must only apply to sludges from dedicated systems. The plain language of 40 C.F.R. § 261.32, adopted by reference at 11 Miss. Admin. Code Pt. 3, R. 1.2, states that "wastewater treatment sludge from the production of toxaphene" is a K041 listed hazardous waste. Noticeably absent from this language are any terms limiting the applicability of the listing to sludges formed from "pure" toxaphene wastewaters or sludges formed solely from wastewater treatment systems dedicated only to managing toxaphene wastewaters. There is no basis for reading the K041 listing as narrowly as Hercules suggests. In fact, the Background Listing Document also cites toxaphene production at the Vertac Chemical Company (also known as Vicksburg Chemical Company) Vicksburg, Mississippi plant in discussing the basis for the listing. Although details regarding the Vicksburg manufacturing process and wastewater treatment system have been redacted from the Background Listing Document, other historical documents available for the Vicksburg facility reveal that Vicksburg did not have a dedicated toxaphene wastewater treatment system, and that several waste streams were, in fact, combined in its wastewater treatment system. *See* Enclosure 3 (September 11, 1974 Vicksburg Chemical NPDES Application). The Background Listing Document itself also recognizes that there were "[v]ariations in wastewater treatment systems [and] in wastewater sources at the two plants."

Background Listing Document at p. 4. Thus, it is contrary to the language of the regulation as well as the Background Listing Document to conclude that only waste streams from dedicated wastewater treatment systems and resultant sludges are covered by the K041 listing.

C. No threshold concentration of toxaphene is required for the K041 listing to apply.

Related to Hercules' argument that the K041 listing only applies to wastewater sludges from pure toxaphene wastewaters treated in dedicated systems is its argument that there is a threshold concentration above which toxaphene must be present before the listing can attach. According to Hercules' interpretation of the Background Listing Document, only those sludges exhibiting the same toxaphene concentrations as wastes from the Brunswick facility would be K041 wastes. *See* Brunini letter at p. 5. This interpretation reads a threshold concentration limit of 10,000 mg/kg into the text of the K041 listing; however, there is nothing in the text of the listing or in the Background Listing Document that supports this interpretation. In fact, Hercules' suggestion that the K041 listing applies only to wastes that meet a concentration threshold is completely at odds with the original concept behind hazardous waste listings which was to "relieve waste generators of much of the testing burden and uncertainties" that might otherwise exist in making hazardous waste determinations. *See* 43 Fed. Reg. 58,946, 58,953 (Dec. 18, 1978).

The Background Listing Document itself recognized that variations in wastewater treatment systems and wastewater sources could result in "different concentrations of toxaphene" in the resultant sludge. *See* Background Listing Document at p. 4. Although data was available for toxaphene concentrations in the sludge at the Brunswick facility at the time of the listing in 1980, no data was available on the amount or concentration of toxaphene in the sludges at the Vertac facility. Notwithstanding this lack of data, there is no question that the K041 listing was intended to cover sludges produced from the Vertac Vicksburg facility.

Hercules' claim that a threshold concentration is required is also undermined by the agencies' regulation of toxaphene at much lower concentrations than 10,000 mg/kg. For example, toxaphene is on the list of toxic pollutants designated pursuant to Section 307(a)(1) of the Clean Water Act, 33 U.S.C. § 1317(a)(1). Pursuant to 11 Miss. Admin. Code Pt. 6, R. 2.2.F., the chronic water criterion for toxaphene in State waters is 0.0002 micrograms per liter (ug/l). Further, the Maximum Contaminant Level (MCL) for toxaphene established pursuant to the Safe Drinking Water Act is 0.003 mg/l, while the universal treatment standard for toxaphene under the RCRA Land Disposal Restriction regulations is 2.6 mg/kg for nonwastewaters. *See* http://water.epa.gov/drink/contaminants/basicinformation/toxaphene.cfm and 40 C.F.R. § 268.48(a). Thus, Hercules' argument that the agencies should only be concerned about toxaphene if the level exceeds 10,000 mg/kg is wholly inaccurate.

Notwithstanding the fact that no threshold concentration is required for the K041 listing to apply, the toxaphene detections found within the IB are indicative of the levels that one might expect to find from toxaphene production that occurred in the 1950s. The highest level of toxaphene found in the IB in recent sampling activities was 75 mg/kg. Sampling performed by the Georgia Environmental Protection Division in 1988 at the Hercules Brunswick facility's equalization basin found toxaphene present in the sludge at a concentration of 80.4 mg/kg. *See* Enclosure 4. Given that toxaphene has a half-life in soil of up to 11 years (*see* http://www.atsdr.cdc.gov/toxprofiles/tp94.pdf), one would expect the concentration of toxaphene in the sludge in the IB to be much lower today than it would have been in the early 1950s when it was first generated. Additionally, the toxaphene sampling Hercules performed in the IB was *after* the solidification process had taken place. *See* Brunini letter at p. 5. As Hercules notes in footnote 3 of the Brunini letter, the stabilization reagents added to the sludge likely decreased the concentration of toxaphene in the sludge by 15-20%. Thus, even though the toxaphene concentration in the IB today is

much lower than the 10,000 mg/kg cited in the K041 Background Listing Document, it is anticipated with toxaphene production at the facility dating back to the 1950s.[6]

D. The text of other listings does not limit the applicability of the K041 listing.

In several places within the Brunini letter, Hercules cites to the text of other listing descriptions and contrasts the wording or applicability of those listings to that of K041 in an attempt to draw meaning from differences in the listings.[7] The EPA has listed hundreds of different wastes under Subpart D of 40 C.F.R. Part 261, each turning on different factors and being promulgated at different times throughout RCRA's thirty-five year history. The text of the K041 listing is very broad. Hercules suggests that we read several limitations into the K041 listing that are simply not there. The EPA has long recognized that "in real-world waste management, many hazardous wastes are mixed with non-hazardous wastes or other hazardous wastes during storage, treatment, or disposal." 45 Fed. Reg. 33,084, 33,095 (May 19, 1980). Courts have recognized that it would be "impractical" and "nearly impossible" for the EPA to bear the "affirmative burden of anticipating and analyzing, in a listing decision, the hazardousness or non-hazardousness [of] every conceivable mixture or derivative that a generator might create." *American Chemistry Council*, 337 F.3d at 1065. As a result, it is inappropriate to read limitations into the K041 listing description where no such limitations were included or intended.

In addition, Hercules' reliance on the 7th Circuit's decision in *U.S. v. Bethlehem Steel*, 38 F.3d 862 (7th Cir. 1994) is misplaced for several reasons. First, in response to the *Bethlehem Steel* decision in 1994, the EPA issued a Memorandum specifically concluding that it had always been the EPA's intent to cover sludges from wastewater mixtures within the scope of several F- and K-listings (including K041). *See* September 13, 1999 Memorandum from David Bussard and David Nielsen to Robert Springer entitled "Sludges from Wastewater Mixtures" (hereinafter, "Sludge Memo"). The EPA acknowledged that "[f]acilities with multiple operations routinely mix their wastewaters prior to treatment, and the Agency intended the listings to cover sludges from these mixtures of wastewaters." *Id.* at p. 1. Second, as noted in the Sludge Memo, the 7th Circuit's decision in *Bethlehem Steel* is not binding outside the 7th Circuit. Third, and most importantly, the *Bethlehem Steel* court did not consider the applicability of the mixture rule, which would most certainly have influenced the court's decision. *See Bethlehem Steel*, 38 F.3d at 870-71. Application of the mixture and derived-from rules to both the incoming wastewater and its resultant sludge further supports the conclusion that the sludge in the IB is a K041 listed waste. *See* discussion in Section I *supra*.

IV. Mississippi is authorized for the mixture and derived-from rules.

As discussed above in Section I, the mixture and derived-from rules are significant components of RCRA's cradle-to-grave hazardous waste management system and serve to clarify what materials must be managed as hazardous waste. The mixture rule acknowledges the reality that hazardous wastes are often mixed with nonhazardous or other wastes, but that such mixing does not necessarily remove the hazardous nature of such wastes. *See* 66 Fed. Reg. 27,266, 27,271 (May 16, 2001). As a result, the mixture and derived-from rules are critical components of the hazardous waste identification and characterization process. The rules were originally promulgated as part of the "base" RCRA program in May 1980 at the same time that K041 was added to the list of hazardous wastes. *See* 45 Fed. Reg. 33,084 (May 19, 1980). Mississippi received authorization for the "base" RCRA program regulations,

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[6] It is also worth noting that the composition of toxaphene in the environment changes over time due to the fact that the congeners of toxaphene degrade at different rates. *See, e.g.,* http://www.atsdr.cdc.gov/toxfaqs. No analysis of this "weathered toxaphene" has been conducted to date of the sludge at the facility.

[7] Hercules contrasts the language of the K041 listing with that of the listings for F026, F032, K174, and F006. *See* Brunini letter at pp. 3, and 4-6.

including the original mixture and derived-from rules, on June 13, 1984. *See* 49 Fed. Reg. 24377 (June 13, 1984). As Hercules notes, the mixture and derived-from rules were vacated by the D.C. Circuit Court in 1991 on procedural grounds; however, the court recognized that the rules were of such import that the EPA should consider reinstating them under the "good cause" exemption of the Administrative Procedure Act, which it did. *See Shell Oil Co. v. EPA*, 950 F.2d 741 (D.C. Cir. 1991); *see also* 57 Fed. Reg. 7628 (Mar. 3, 1992) (reinstating the mixture and derived-from rules on an interim basis pending full notice and comment).

When the EPA reinstated the mixture and derived-from rules in 1992, the previously authorized state mixture and derived-from rules, which were not vacated by the federal court, were already within the scope of the federal RCRA program. As a result, the EPA considered the 1992 rule to be "optional" for states previously authorized for the mixture and derived-from rules, meaning that because the 1992 rules did not make any substantive changes to the federal regulations, previously authorized states were not required to seek authorization for the reissuance. *See* Revised Checklist 117A Summary (OSWER Directive No. 9541.0020). Mississippi had already received authorization for the original mixture and derived-from rules. As a result, it did not need to seek and receive authorization for the reinstatement. [8]

Ultimately, the EPA finalized the retention of the mixture and derived-from rules on May 16, 2001. *See* 66 Fed. Reg. 27,266 (May 16, 2001). These rules were upheld by the D.C. Circuit as reasonable interpretations of the Agency's statutory authority to regulate hazardous waste from cradle-to-grave. *See American Chemistry Council*, 337 F.3d at 1065. At the time of the finalization of the rules, the EPA again confirmed that states that had already received authorization for the mixture and derived-from rules did not need to obtain authorization for those rules again. *See* 66 Fed. Reg. at 27,293 (also confirming that the rules were already in effect in those authorized states). However, the 2001 rule also made certain revisions to those rules, which states were required to adopt and receive authorization for before they would go into effect in the states. The State of Mississippi adopted the 2001 final rules, with their associated revisions, and received federal authorization for such rules on August 4, 2008. *See* 73 Fed. Reg. 45170 (Aug. 4, 2008).

Thus, despite Hercules' contention to the contrary, the mixture and derived-from rules are currently valid and effective components of the State of Mississippi's authorized hazardous waste program. For purposes of characterization of the sludge in the IB for disposal purposes, there is no question that the current mixture rule applies. The fact that K041 sludges may have been mixed with other sludges through normal operation of the IB does not negate the K041 listing. In fact, such mixing results in the entirety of the IB sludge carrying the K041 listing under the mixture rule.

[8] Hercules cites Mississippi's pending request to be authorized for Checklist 117A (the 1992 mixture and derived-from rule reinstatement) as proof that Mississippi requires authorization for that Checklist in order for the EPA to apply the mixture or derived-from rules in the State. Although Mississippi's Program Revision Application, submitted to the EPA in September 2014, included a request to be authorized for Checklist 117A, along with several other historic checklists, this submission was solely made in an effort to pick up several optional checklists for which Mississippi had not previously sought authorization. As Hercules correctly notes, the EPA has not processed this pending application. Authorizing Checklist 117A now, especially given Mississippi's authorization for the final mixture and derived-from rules, is unnecessary and would likely create unnecessary confusion on the part of the regulated community. As a result, the EPA and MDEQ are discussing the most appropriate way of addressing the pending authorization application for Checklist 117A.

V. Conclusion

Given the recent sampling results showing toxaphene in the IB, as well as documentary evidence of historic toxaphene production at the facility, the EPA and MDEQ have concluded that the sludge at the Hercules facility does, in fact, carry the K041 listing. Although Hercules argues that it did not have "fair notice" that the sludges would be considered to be K041 wastes, the agencies' position on the applicability of the listing and the operation of the mixture and derived-from rules is not novel and is consistent with RCRA's purpose to regulate hazardous waste from cradle to grave. In addition, Hercules, as the owner and operator of the facility, is in the best position to assess available information regarding wastes generated at the facility and it is Hercules' own documents that illustrate that toxaphene wastewaters were sent to the IB and resulted in the creation of the K041 sludge, as confirmed by the presence of toxaphene in the recent IB sampling. We encourage Hercules to contact the EPA and MDEQ to discuss how we might best proceed in light of this determination.

Sincerely,

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U.S. Environmental Protection Agency, Region 4

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Senior Attorney
Mississippi Department of Environmental Quality

Enclosures

cc: Tim Hassett, Hercules (via email)
John Ellis, ARCADIS (via email)

Enclosure 1

MANUFACTURE OF TOXAPHENE

Date	Toxaphene Made Pounds	Materials Used - Pounds: Camphene	Chlorine	Carbon Tetra-chloride	Ortho-leum	Propy-lene Oxide	Benzoyl Chloride
1951	14,511,087	5,397,407	20,250,000	1,815,569	1,700	36,600	70,462
1952							
Jan.	2,345,234	826,600	3,084,000	245,567	200	4,440	14,178
Feb.	2,515,345	862,551	3,340,000	275,063	100	5,400	14,335
Mar.	2,626,691	870,865	3,440,600	259,273	200	5,040	13,642
Apr.	2,487,144	871,397	3,340,000	268,470	150	5,220	14,710
May	1,885,720	647,591	2,620,000	183,988	150	4,500	12,038
June	764,564	255,181	950,000	139,563	50	2,160	5,082
July	-	-	-	-	-	-	-
Aug.	-	-	-	-	-	-	-
Sept.	-	-	-	-	-	-	-
Oct.	-	-	-	-	-	-	-
Nov.	-	-	-	-	-	-	-
Dec.	-	-	-	-	-	-	-
Total	12,624,698	4,334,185	16,774,600	1,371,924	850	26,760	73,985

Date	Materials Used - Pounds - Cont'd: Sodium Peroxide	Benzoyl Peroxide	Lime	Lime Rock	Calcium Chloride	Caustic	Sodium Bisulfite
1951	23,675	3,200	4,044,060	5,064,400	4,771	-	50
1952							
Jan.	7,125	200	497,500	744,400	400	-	-
Feb.	5,200	150	347,200	825,800	400	-	-
Mar.	4,700	100	597,500	882,300	200	-	50
Apr.	4,900	100	719,800	1,620,500	-	-	-
May	4,800	350	522,500	1,434,400	460	-	-
June	1,600	300	161,250	374,800	1,870	-	-
July	-	-	-	-	-	-	-
Aug.	-	-	-	-	-	-	-
Sept.	-	-	-	-	-	-	-
Oct.	-	-	-	-	-	-	-
Nov.	-	-	-	-	-	-	-
Dec.	-	-	-	-	-	-	-
Total	28,325	1,200	2,845,750	5,782,200	3,330	-	50

Enclosure 2

Hattiesburg, Mississippi
April 30, 1952.

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Cooper
RI Htbg. 93 (N.S.) - Hicks - CHLORINATED CAMPHENE - Manufacturing Problems.

N.S. - Process Service.

Summary of April Production. Problems from Oxidized Pinene-24n

DIGEST

All of the 2,487,141 pounds of toxaphene produced during April was on-grade (Class 0). The average chlorine content of the toxaphene drummed was 67.5%. The chlorine efficiency dropped to 87.4%. The CCl_4 loss was 0.108 pound per pound of toxaphene.

HCl production was 988,300 pounds of 20.1° Be'. acid, which is 24.6% of theory and 27.3% of the recoverable acid. This production includes only the acid shipped or retained for shipment.

Plant observations and laboratory data are included revelant to the February period when low softening point toxaphene was produced. The reasons for the low softening point are not definitely known. Several factors may have contributed to this off-grade production; of these, no single reason or group of reasons can be stated as the actual cause of the off-grade production.

A toxaphene ditch water sample was composited for Dr. Renn at Johns Hopkins University. The fact that it was collected at the toxaphene area should be considered in connection with any test data obtained on the sample, because toxaphene waste water would normally have passed through several additional treating stages prior to leaving the plant and its final entry into the Bouie River.

Pinene deteriorates rapidly during storage due to the formation of oxidation products. These oxidation products inhibit the TiO_2 catalyzed isomerization to camphene. Therefore, pinene for the camphene process must be used very soon after distillation to obtain short isomerization times. Reworking old pinene is both costly and time-consuming. Laboratory tests have shown that the storage time can be greatly increased with only a small change in the isomerization time, when 0.001% hydroquinone, an antioxidant, is added to fresh pinene. In these tests, fresh wood pinene isomerized in 2.2 hours, the isomerization time increased to 5 hours during 5 days storage, and to 55 hours during 20 days storage. A portion of the same pinene, but treated with 0.001% hydroquinone at the beginning of the storage test, isomerized in only 5 hours after 20 days storage.

Camphene from hydroquinone-treated pinene was of good quality and produced toxaphene meeting all specifications.

Tests are being made to determine the effectiveness of hydroquinone when used with sulfate pinene.

A series of TiO_2 catalysts, having different final acetic acid contents, was prepared by dividing a large batch of catalyst

after the initial acidification step. It was observed that the time required to isomerize pinene to camphene decreased with decreasing acid content to about 0.06% acetic acid; below which no significant change in isomerization time was detected.

All camphene was analyzed on a routine basis by infrared spectrophotometry.

Discussion.

Production.

Toxaphene production for April was 2,487,141 pounds; all of the production was of Class O quality. The production for this month was lower than during March because of the limited amount of chlorine available. The average chlorine content of the toxaphene drummed was 67.5%. The CCl_4 loss was 0.108 pound per pound of toxaphene. The chlorine efficiency for the month was 87.4%. This is lower than during previous months.

Table 1 shows a summary of the toxaphene production and quality on a monthly basis.

The HCl production was 988,300 pounds of 20.1° Be. acid, which was 24.6% of theory and 27.3% of the recoverable acid. This production includes only the acid shipped or retained for shipment. Acid neutralized and dumped to the ditch because of the tank car shortage is not included.

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RI Htbg. 93 (N.S.) - Cooper Hicks

April 30, 1952.

TABLE 1

SUMMARY OF TOXAPHENE PRODUCTION AND QUALITY

Date	Pounds Produced	Classification of Salable Toxaphene							Per cent Off-Grade	Average Chlorine Content	Chlorine Efficiency
		0	1	2	3	4	5	6 Total			
1951[1].	14,511,078	96.27%[2].	1.73%[2].	0.63%[2].	0.41%[2].	0.05%[2].	-	- 99.24%[2].	0.76%[2].	67.9%[2].	85.3%[2].
1952											
Jan.	2,352,520	97.27%	0.53%	-	-	-	-	- 97.80%	2.20%	67.7%	90.1%
Feb.	2,515,345	95.16%	0.22%	-	-	-	-	- 95.38%	4.62%	68.1%	89.5%
Mar.	2,626,691	99.59%	-	-	-	-	-	- 99.59%	0.41%	68.0%	90.5%
April	2,487,141	100.00%	-	-	-	-	-	- 100.00%	-	67.5%	87.4%

Notes:

1. Toxaphene plant in operation eight months, May through December, during 1951.
2. Weighted average based on toxaphene.

Toxaphene - Analysis of Materials Removed from the Toxaphene Plant During the February 9-15th Period of Off-Grade Production.

During February a period was experienced when the toxaphene production was below grade in softening point. The reasons for the low softening point are not definitely known. Several factors may have contributed to this off-grade production; of these, no single reason nor group of reasons can be stated as the actual cause of the off-grade production. Observations on plant and laboratory information pertinent to this period of production are given below.

1. Plant Process.

(a) The camphene used was slightly below average in the ratio of camphene to tricyclene.

(b) Iron addition to the prechlorination stage and even to the final reactors did not show the normal influence on softening point.

(c) The characteristics of the chlorination reaction mixture appeared normal (with a high softening point) until it entered "C" reactor. Therefore, "C" reactor was drained to remove the low melting product and fresh feed from "B" reactor then entered "C". The characteristics of this fresh reaction mixture continued to appear normal in "C" reactor until the chlorine content increased to 67.5-68.0%. At this point the softening point dropped sharply to the 45° range.

(d) Batchwise chlorination in "A" and "B" chlorinators produced on-grade toxaphene. Batchwise production in "C" was not attempted.

(e) "C" chlorinator was opened for examination. Considerable solid material had accumulated on the bottom of the reactor and nine sparge arms were broken. The solid material was cleaned out but the broken sparge arms were not replaced. Following this cleaning, "C" chlorinator was returned to service and the toxaphene produced was of high softening point.

(f) "C" Reactor was opened recently to replace the broken sparge arms. "A" and "B" reactors were opened at the same time. Considerable solid material had accumulated in these reactors. The amount of solid material present in "C" reactor was almost equal to that found during the February cleaning. The solids were removed from all three reactors. Toxaphene produced immediately prior to this recent cleaning was on-grade with no indication of any impending difficulty with the softening point specification.

2. Laboratory Findings.

(a) H111-116. The camphene used in the plant at the time of the low softening point product was chlorinated in the laboratory to toxaphene having a softening point of 98°C. This camphene was somewhat inferior to the usual camphene and had the following composition as determined by infrared spectrophotometry:

Camphene	75%
Tricyclene	19%
a-Pinene	0.6%
Bornylene	0.6%

(b) H111-117. Chlorination in the laboratory of a sample withdrawn from "A" reactor (4 P.M., 2-11-52) produced

on-grade toxaphene.

(c) H112-18, 19, 32. Since over-chlorination was one possible explanation for the low softening point, increments of a sample from "B" reactor were chlorinated to increasingly higher chlorine contents, indicating that the softening point decreased with increasing chlorine content as shown below:

Per cent Chlorine in Product	Softening Point
67.3%	106.5°C.
68.5%	98.0°C.
68.7%	96.0°C.
69.4%	86.0°C.

Even though the softening point decreased, it was well within the specifications at as high as 69.4% chlorine in product.

(d) H114-77. A camphene chlorination was performed to which solid material from the plant system (5% by weight, based on toxaphene produced) was added to the reaction mixture following the prechlorination stage. After the chlorination was completed, half the reaction mixture was filtered, allowing the salt to accumulate in the unfiltered portion. Both portions were then sparged in the usual manner to remove CCl_4. The toxaphene from both portions had high softening points. The softening points were 100°C. with the salt removed and 106°C. with salt present.

(e) Spectrographic analysis for metals present in toxaphene samples taken during high and low softening point production showed no significant difference in the products. See Table 2. Also, no difference was observed in the samples removed from "B" and "C" chlorinators.

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TABLE 2

ANALYTICAL RESULTS[1] ON TOXAPHENE SAMPLES

Sample Designation	Description	Analytical Results Metals Present in P.P.M. (by spectrographic analysis) Cu	Fe	Ni, Cr, Pb	Al, Ca	Mg	Mn	Zn
H112-43-1	Toxaphene, m.p. 90°C.	0.8	<2	*4	*10	<10	*0.4	*30
H112-43-2	Toxaphene, m.p. 80°C.	3	<2	*4	*10	<10	*0.4	*30
H112-43-3	Toxaphene, m.p. 39°C.	0.8	*2	*4	*10	<10	*0.4	*30
H112-43-4	Toxaphene, m.p. 42°C.	0.8	<2	*4	*10	<10	*0.4	*30
H112-43-5	Chlorinated camphene soln. from "B" chlorinator	*0.5	*2	*4	*10	<10	*0.4	*30
H112-43-6	Chlorinated camphene soln. from "C" chlorinator	*0.5	*2	*4	*10	<10	*0.4	*30

< = Less than, but detected

* = Less than, and not detected.

[1] Data obtained at Experiment Station. See letter Propst, M. E., to Hicks, H. E., dated March 31, 1952.

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Table 2 - Cont'd

Sample Designation	Description	Analytical Results
H112-43-7	Deposit on "C" chlorinator wall near bottom, before washing	Particles of carbon were observed visually. Microscopic examination indicated the material to be largely NaCl dispersed in toxaphene, with a very small amount of fibrous material, probably asbestos. A small amount (order of 1%) of other crystalline material was observed, which appeared similar to quartz. Infrared absorption indicated that the CS_2-soluble portion was mostly toxaphene with CCl_4 a minor constituent. Weak absorption in the region of 9.7-9.9 mu was not identified. Acetone washings of the CS_2-insolubles gave a slight yellow color but had no detectable infrared absorption. A Fractol mull of the insoluble residue had no identifiable absorption.
H112-43-8	Deposit on "C" chlorinator wall near bottom, after washing	Sample was similar to 43-7. No difference was noted in the two samples.
H112-43-9	Material from "C" chlorinator reflux line.	Microscopic examination indicated a small amount of NaCl present. A noncrystalline yellow matrix was identified. A large amount of fibrous material present was identified as asbestos. The sample was largely insoluble in organic solvents, while a small portion (NaCl) was soluble in water. A Fractol mull of the sample showed little infrared absorption, none of which was similar to toxaphene or BHC. An -OH band was observed and a band at 9.80 mu suggested the possibility of its presence as a primary alcohol.
H112-43-10	Material from "C" chlorinator reflux sight glass.	Microscopic analysis indicated NaCl with only minute impurities. The sample was almost completely water-soluble.

Table 2 - Cont'd

Results of Spectrographic Analysis for Metals (%) (approximations)

H112-43-	7	8	9	10
Mg	0.2	0.2	>0.3	0.08
Mn	>0.001	>0.001	>0.001	>0.001
Pb	0.008	0.008	*0.002	0.002
Si	0.02	0.02	>0.02	0.003
Fe	0.015	0.02	>0.02	0.003
Cr	0.002	0.002	0.002	0.0005
Ni	>0.02	>0.02	0.01	0.002
Al	0.015	0.015	0.03	*0.004
Ca	0.01	0.01	0.01	0.003
Cu	>0.01	>0.01	0.001	<0.0002
Zn	*0.006	*0.006	*0.006	*0.006
Na	Major	Major	Major	Major

< = Less than, but detected.

* = Less than, and not detected.

(f) Analysis of the deposit from "C" chlorinator showed the material was principally sodium chloride and chlorinated camphene, with minor amounts of carbon particles, asbestos, and an unidentified noncrystalline material, probably Haveg binder. See Table 2.

(g) The crystals from the "C" chlorinator reflux sight glass were almost entirely sodium chloride. Also present were asbestos in appreciable quantities and an unidentified noncrystalline material. Infrared absorption indicated the present of an -OH group, probably in the form of a primary alcohol.

In addition to the above factors, it is possible that localized overheating caused by poor heat transfer through the solids adhering to the reactor wall contributed to the off-grade production.

In consideration of all of the above information, it is extremely difficult to select a definite reason for the production of low-melting toxaphene during this (February) period.

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Toxaphene - Ditch Water Sample.

A 5-gallon sample of ditch water from the toxaphene area was composited from six daily samplings during the week of April 23-30, for shipment to Dr. Renn at Johns Hopkins University, Baltimore, Maryland. This ditch water was collected through a manhole in the camphene area and represents the waste water leaving the toxaphene area. This waste water, however, is quite different from the waste actually leaving the plant. The toxaphene waste water, after passing the above manhole, passes through (1) a lime-

stone basin, (2) an impounding (sedimentation) basin, where it is combined with and diluted by other plant waste water, (3) a pH recording and controlling station where low pH effluent is adjusted to a value of 7, and finally (4) through a ditch where mixing and aeration occur prior to entry into the Bouie River.

The fact that this toxaphene waste water sample for Dr. Renn was taken at an intermediate point in the plant's waste water treating system should be considered in connection with any test work performed on this sample.

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Crude Turps - Accumulation in Topping Column.

A sample of scale removed from the sulfate turps topping column was analyzed at the Experiment Station. Their results(1) indicated the sample contained a considerable portion of magnetic material. Combustion analysis indicated:

Carbon	11.7, 13.1%
Hydrogen	1.53, 1.73%
Sulfur	2.2 , 2.5%
Ash (residue from combustion)	76.7, 79.9%

The ash was reddish brown.

Microscopic analysis showed a small amount of noncrystalline material, apparently polymeric. X-ray diffraction identified a mixture of Fe_2O_3 and Fe_3O_4. A Fractol mull of the sample showed no identifiable infrared absorption. However, polymeric material expected to be present might well have weak, non-specific absorption which would not show up in these spectra. Spectrographic analysis for metals indicated the following were present:

(1) ltr. Propst, M.E., to Hicks, H.E., dated March 31, 1952.

Magnesium	Less than 0.04%
Manganese	More than 0.02%
Lead	Less than 0.02% and not detected.
Silicon	More than 0.3%
Iron	More than 0.3%
Chromium	0.02%
Nickel	Less than 0.008% and not detected.
Aluminum	0.02%
Calcium	0.02%
Copper	More than 0.2%
Zinc	Less than 0.03% and not detected.
Sodium	Not Detected.

- - - - -

Pinene - Addition of Antioxidant to Improve Storage Characteristics.

Pinene deteriorates during storage due to the formation of oxidation products. These oxidation products inhibit the TiO_2 catalyzed isomerization of pinene to camphene. This places a limitation on the length of time pinene can be stored without seriously impairing the rates obtained during conversion to camphene. It has also been necessary to redistill a condiderable amount of pinene which had gone "stale" during storage, in order to obtain a suitable material for use in the camphene process. This procedure required extra still time and is not economical for several reasons.

The addition of an antioxidant, hydroquinone, to pinene was tested as a possible means of preventing this deterioration during storage. In this test, fresh wood pinene (W-27, April 2, 1952) was stored in two 5-gallon glass carboys. Hydroquinone (0.001% by weight) was added to the contents of one of the carboys; the pinene in the second carboy served as the control and therefore was not treated. It was observed that hydroquinone was not soluble to the extent of 0.001% in pinene.

Pinene from each storage sample was removed and isomerized periodically to determine the time required for isomerization.

Results are shown in Table 3. The data are plotted on Figure 1.

The results showed that addition of hydroquinone to fresh pinene definitely improved the storage characteristics of pinene as measured by the isomerization test. After twenty days storage, the isomerization time had increased to five hours for pinene treated with hydroquinone, whereas the control pinene required fifty-five hours to complete the isomerization.

Addition of hydroquinone to "stale" pinene improved the isomerization rate. This treatment was not so beneficial as introducing the additive at the beginning of the storage period.

Pinene which had been stored one month with hydroquinone present was isomerized. This crude camphene was fractionated in the laboratory, simulating plant conditions. The camphene cut from this fractionation was of good quality, as shown below:

Distillation Serial No. 318

Light end cut	1.1%
Camphene cut	76.0%

Infrared analysis of camphene cut:

Camphene	80%
Tricyclene	18%
α-pinene	Trace.

On chlorination, this camphene produced toxaphene meeting all specifications. Results of analysis follow:

Chlorine content	68.1%
Softening point	103°C.
Acidity (HCl)	222 p.p.m.
Color	2.0
Deo-Base cloud test	Clear
Stability	3.0

Tests are being made to determine the effectiveness of hydroquinone when used with sulfate pinene.

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TABLE 3

EVALUATION OF HYDROQUINONE AS A PINENE ADDITIVE

TO IMPROVE STORAGE CHARACTERISTICS

Notebook Reference	Date	Days Storage	TiO_2 Catalyst Batch No.	TiO_2 Catalyst Amount	Isomerization Time, Hrs. With No Additive	Isomerization Time, Hrs. With Hydroquinone
H114-51	4/ 2/52	0	35	0.25%	2.2	-
H114-52	4/ 3/52	1	35	0.25	3.0	-
H114-53	4/ 3/52	1	35	0.25	-	2.5
H114-62	4/ 7/52	5	35	0.25	4.5	-
H114-63	4/ 7/52	5	35	0.25	-	3.5
H114-85	4/15/52	13	35	0.25	-	3.5
H114-86	4/15/52	13	35	0.25	25	-
H114-98	4/22/52	20	35	0.25	-	4.75
H114-99	4/22/52	20	35	0.25	±55*	-
H116-10	4/28/52	26	35	0.25	-	7

*Hydroquinone was added to another portion of this pinene which had been stored 20 days without any additive. The hydroquinone-pinene solution was allowed to stand 16 hours prior to isomerization. The isomerization rate was improved by this treatment, as the isomerization time was reduced from ± 55 to 30 hours, as below:

H114-100	4/22/52	20	35	0.25	30*

The acid number was determined on the storage samples after 28 days storage. Values of 0.005 and 0.002 were obtained for the control and the treated pinene, respectively.

TiO_2 Activation.

A study of the initial acidification step in the preparation of TiO_2 catalyst used in camphene manufacture has shown that this step is of major importance in the production of high activity catalyst. It was observed (RI Htbg. 93 N.S.-Hicks, Feb. 29, 1952) that catalyst activity increased rapidly with increasing initial acetic acid concentration, even though the initial caustic treatment and final acetic acid concentrations were the same. This work was extended to determine the effect of the final acetic acid concentration on the resulting catalyst activity.

A large batch of TiO_2 acid cake was treated with 50% caustic and washed in the usual manner to remove impurities. The washed slurry was then acidified with glacial acetic acid to obtain an initial acetic acid concentration of 1.386%. This acidified slurry was divided into aliquots which were washed individually to obtain catalyst with various final acetic acid contents, ranging between 1.386% and 0.003%. The activity of these catalyst was determined on wood pinene. The time required to isomerize the pinene to camphene with this series of catalysts is shown below (see also Table 4).

Final Acetic Acid Concentration	Isomerization Time in Hours (0.25% TiO_2 Catalyst present)
1.386%	± 64
0.317	± 33
0.228	7, 11
0.056	2.5
0.041	3
0.023	3
0.014	3, 2
0.003	3.5

The isomerization times obtained using this series of catalysts indicated that the isomerization time decreased as the

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TABLE 4

EFFECT OF THE FINAL ACETIC ACID CONCENTRATION ON THE ACTIVITY OF TiO_2 CATALYST

CATALYST PREPARATION					PINENE ISOMERIZATION		
Notebook Reference	Acetic Acid Content Initial	Acetic Acid Content Final	Solids Content	Sp.Gr. of Slurry	Notebook Reference	Amount of Catalyst Used (Solids Basis)	Isomerization Time in Hours
H112-81	1.386	1.386			H114- 6	0.25%	± 64
H114-68	1.386	0.317	19.8	1.111	H114-75	0.25	± 33
H114- 2	1.386	0.228	15.6	1.090	H114-30	0.25	11
					H114-12	0.25	7
H114- 1	1.386	0.056	15.3	1.097	H114-15	0.25	2.5
H114- 4	1.386	0.041	18.3	1.093	H114-19	0.25	3
H114-27	1.386	0.023	15.3	1.097	H114-36	0.25	3
H114- 3	1.386	0.014	14.8	1.095	H114-18	0.25	3
					H114-16	0.25	2
H114-26	1.386	0.003	34.2	1.222	H114-71	0.25	3.5

acid content decreased until the final acetic acid concentration was about 0.06%. At 0.06% and lower final acetic acid concentrations, no significant change in activity was observable.

Very long settling times were experienced during the final washing of catalysts having very low acetic acid concentrations.

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Pinene - Isomerization.

The activity of each batch of titanium dioxide catalyst prepared in the plant was determined in the laboratory. Also, a sample from each tank car of pinene received from the still house was isomerized in the laboratory prior to use in the plant. These isomerizations are shown in Table 5.

TABLE 5

PINENE ISOMERIZATIONS - LABORATORY

Notebook Reference	Pinene Identification	TiO_2 Catalyst Batch No.	TiO_2 Catalyst % Solids	TiO_2 Catalyst % Acetic Acid	Isomerization Time (Hours)
H114-47	HPCX-6090, Sampled 3/31/52	39*	4.8	0.053	10.5
H114-50	HPCX-6090 " 4/ 1/52	39	10.9	0.053	12
H114-56	GATX-206 " 4/ 3/52	39	10.9	0.053	14
H114-60	HPCX-6090 " 4/ 5/52	39	10.9	0.053	8
H114-61	HPCX-6090 same as above	40	11.5	0.043	7
H114-64	TC-19 Sampled 4/ 7/52	40	11.5	0.043	3
H114-67	GATX-206 " 4/ 8/52	40	11.5	0.043	2.5
H114-69	HPCX-6090 " 4/ 9/52	40	11.5	0.043	2.8
H114-78	GATX-206 " 4/10/52	40	11.5	0.043	4
H114-79	HPCX-6090 " 4/11/52	40	11.5	0.043	3
H114-80	GATX-206(1) " 4/12/52	41	11.9	0.046	5
H114-81	GATX-206 " 4/14/52 same as above	41	11.9	0.046	3.8
H114-82	GATX-206 same as above	40	11.5	0.043	2
H114-83	HPCX-6090, Sampled 4/14/52	41	11.9	0.046	6
H114-89	GATX-206 " 4/15/52	41	11.9	0.046	5.5
H114-90	HPCX-6090 " 4/17/52	41	11.9	0.046	4
H114-95	TC-18 " 4/21/52	41	11.9	0.046	8
H114-94	TC-18 " 4/21/52	42	11.8	0.033	7
H114-96	HPCX-6090 " 4/21/52	42	11.8	0.033	5
H116- 2	HPCX-6090 " 4/22/52	42	11.8	0.033	8(2)
H116-3	HPCX-6032 " 4/23/52	42	11.8	0.033	9(2)
H116-4	HPCX-6090 " 4/23/52	42	11.8	0.033	9(2)
H116-7	HPCX-6032 " 4/25/52	42	11.8	0.033	10(2)
H116-8	HPCX-6090 " 4/26/52	42	11.8	0.033	6
H116-9	TC-18 " 4/28/52	43	11.0	0.041	23(3)
H116-16	HPCX-6023 " 4/28/52	43	11.0	0.041	2.5

Notes:

(1) This pinene was stored 40 hours in a glass bottle prior to isomerization, whereas the pinene used in H114-81 had been stored in the tank car for the same period prior to isomerization.

(2) This pinene contained some reworked pinene which had been in storage for several months.

(3) This was sulfate pinene which had been in storage for several weeks.

Camphene - Scale in Camphene Still.

A sample of scale removed from the camphene still was analyzed at the Experiment Station. Their results (1) indicated the sample contained a considerable portion of magnetic material. Combustion analysis indicated:

Carbon	50.0, 51.1%
Hydrogen	4.37, 4.54%
Sulfur	0.3, 0.3%
Ash (Residue from combustion)	31.9, 33.7%

The ash was grey-black.

Microscopic analysis indicated a large portion was non-crystalline material, apparently polymeric. X-ray diffraction identified a mixture of Fe_2O_3, Fe_3O_4, and CuO. A Fractol mull of the sample gave no identifiable infrared absorption. Spectrographic analysis for metals indicated the following were present:

Magnesium	Less than 0.04%
Manganese	Greater than 0.02%
Lead	0.2%
Silicon	Greater than 0.3%
Iron	Greater than 0.3%
Chromium	0.02%
Nickel	Less than 0.008% and not detected.
Aluminum	0.4%
Calcium	0.02%
Copper	Greater than 0.2%
Zinc	2.0%
Sodium	Not detected.

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Camphene - Analysis.

Camphene prepared for the toxaphene plant was analyzed by infrared spectrophotometry. The results of these analyses are shown on Figure 2.

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(1) ltr. Propst, M.E., to Hicks, H. E., dated March 31, 1952.

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FIGURE 2

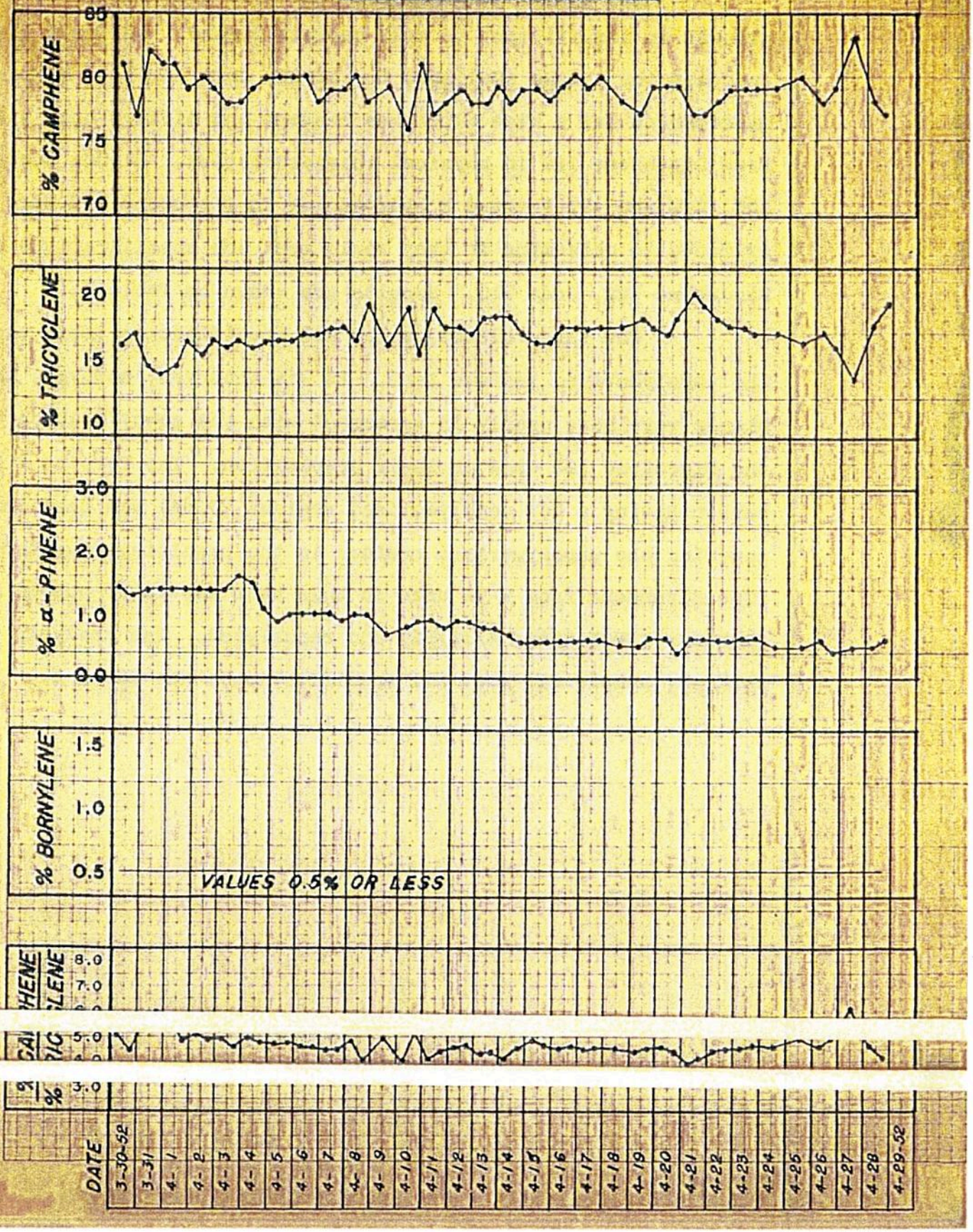
APRIL 30, 1952.

HERCULES POWDER COMPANY

QUALITY CONTROL CHART

HATTIESBURG PLANT

CAMPHENE SAMPLES



Terpene B Hydrocarbon - Use in Dipentene.

The amount of Dipentene 122, Code 210, available at present is insufficient to meet current demand. We would like to increase the volume available to satisfy this demand. Terpene B hydrocarbon has a high dipentene content and differs only slightly from Dipentene 122 in physical characteristics. Blends containing 10%, 20% and 40% Terpene B hydrocarbon in Dipentene 122 were examined to determine if they would meet the specifications for Dipentene 122, Code 210. Results are shown on Table 6.

The three blends passed all specification tests except the unpolymerized residue test. It was observed, however, that the blends were more nearly in agreement with the specification than the Dipentene 122 used in their preparation. The Terpene B hydrocarbon having a low unpolymerized residue (0.8%) was beneficial in lowering the unpolymerized residue in the resulting dipentene blend. (The dipentene from W-94 used in this test was later pumped to a tank car (HPCX-8063; 4/12/52) and reanalyzed. The analysis on the tank car sample did meet all specifications; see Table 6.

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RI Htbg. 93 (N.S.) - Hicks

April 30, 1952.

TABLE 6

ANALYSIS OF TERPENE B HYDROCARBON-DIPENTENE 122 BLENDS

	Dipentene 122 Code 210 Specification Max.	Min.	Tank Car[3] HPCX-8063 4/12/52	D[1]	A	B	C	E[2]
Composition of Blend								
Dipentene 122[1]			100%	100%	90%	80%	60%	-
Terpene B Hydrocarbon[2]			-	-	10%	20%	40%	100%
Properties								
A.S.T.M. Distillation, °C.								
1st Drop			174.0	174.5	174.2	175.0	175.8	179.4
1%								
5%	177.0	173.0	175.2	176.2	175.2	176.0	176.9	180.2
10%	177.0	174.0	175.5	176.5	176.0	177.0	177.0	180.6
20%			176.2	177.0	176.5	177.2	178.0	181.0
30%			176.5	177.5	177.0	177.6	178.3	181.1
40%			177.0	177.7	177.1	178.0	178.5	181.5
50%			177.4	178.2	177.3	178.2	179.0	181.7
60%			177.8	178.5	177.8	178.8	179.2	182.0
70%			178.0	178.8	178.2	179.1	179.9	182.4
80%			178.5	179.2	178.9	179.8	180.2	183.0
90%			179.5	180.5	180.0	180.0	181.5	184.0
95%			181.0	181.5	181.5	181.9	183.0	185.1
97%	188.0	-						
% Over at 188°C.			98.5	97.6	97.8	97.8	97.5	97.5
Specific Gravity at 15.6°C.	0.854	0.845	0.8519	0.8504	0.8508	0.8511	0.8519	0.8538
H_2SO_4 Test	2.0%		2.0	2.8	2.8	2.6	2.4	0.8
Ref. Index at 20°C.	1.477	1.472	1.4741	1.4739	1.4745	1.4750	1.4764	1.4801
Color - P.E.	0.4		0.35	0.25	0.25	0.25	0.25	0.20
Aniline Point			-	7.0	6.0	7.0	6.0	8.0
Appearance	Clear and free from suspended matter		OK	OK	OK	OK	OK	OK

NOTES: (1) Dipentene W-94 (4/8/52). (2) Terpene B hydrocarbon W-28 (4/8/52). (3) Tank car HPCX-8063 (4/12/52) was filled with Dipentene using the contents of W-94 plus about 500 gal. (±6% dipentene from another tank.

It was concluded that up to 40% terpene B hydrocarbon could be added to Dipentene 122 meeting Code 210 to form a blend which would also meet Code 210 specifications. Blends containing more than 40% terpene B hydrocarbon were not examined.

Samples of the Dipentene 122 and Terpene B hydrocarbon-Dipentene 122 blends containing 20% and 40% Terpene B hydrocarbon were sent to Mr. F. W. Volk for observation(1).

Hughe D. Cooper, May 23, 1952.
Hughe D. Cooper.

Harold E Hicks, May 23, 1952
Harold E. Hicks.

READ AND UNDERSTOOD BY:

R. J. Bynkir
CHIEF CHEMIST.
May 23, 1952

(1) Letter H. G. Holleman to F. W. Volk, April 25, 1952, "Use of Terpene B Hydrocarbons in Dipentene 122".

Enclosure 3

FORM APPROVED
OMB No. 158-R0100

FOR AGENCY USE
MSC02799S

NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM
APPLICATION FOR PERMIT TO DISCHARGE WASTEWATER

STANDARD FORM C – MANUFACTURING AND COMMERCIAL

SECTION I. APPLICANT AND FACILITY DESCRIPTION

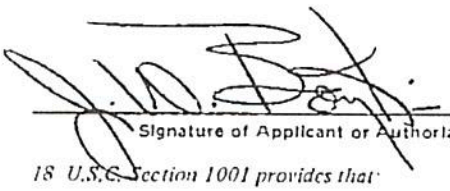
Unless otherwise specified on this form all items are to be completed. If an item is not applicable indicate 'NA.'

ADDITIONAL INSTRUCTIONS FOR SELECTED ITEMS APPEAR IN SEPARATE INSTRUCTION BOOKLET AS INDICATED. REFER TO BOOKLET BEFORE FILLING OUT THESE ITEMS.

Please Print or Type

1. Legal Name of Applicant (see instructions)	101	VICKSBURG CHEMICAL CO.
2. Mailing Address of Applicant (see instructions) Number & Street	102a	P. O. BOX 3
City	102b	Vicksburg
State	102c	Mississippi
Zip Code	102d	39180
3. Applicant's Authorized Agent (see instructions) Name and Title	103a	Jack W. Boykin Vice President
Number & Street Address	103b	P. O. Box 3
City	103c	Vicksburg
State	103d	Mississippi
Zip Code	103e	39180
Telephone	103f	601 (Area Code) 636-1231 (Number)
4. Previous Application If a previous application for a National or Federal discharge permit has been made, give the date of application. Use numeric designation for date.	104	NONE (YR MO DAY)

I certify that I am familiar with the information contained in this application and that to the best of my knowledge and belief such information is true, complete, and accurate.

Jack W. Boykin Printed Name of Person Signing	102e	Vice President-Organic Manufacturing Title
 Signature of Applicant or Authorized Agent	102f	1974 9 11 (YR MO DAY) Date Application Signed

18 U.S.C. Section 1001 provides that:

Whoever, in any matter within the jurisdiction of any department or agency of the United States knowingly and wilfully falsifies, conceals or covers up by any trick, scheme, or device a material fact, or makes any false, fictitious or fraudulent statement or representation, or makes or uses any false writing or document knowing same to contain any false, fictitious or fraudulent statement or entry, shall be fined not more than $10,000 or imprisoned not more than five years, or both.

FOR AGENCY USE

Received ___ ___ ___ (YR MO DAY)

OFFICE: ___ EPA Region Number
___ State

FOR AGENCY USE
MSCD279995

5. Facility/Activity (see instructions) Give the name, ownership, and physical location of the plant or other operating facility where discharge(s) does or will occur.

Name	105a	VICKSBURG CHEMICAL CO. VICKSBURG MISSISSIPPI PLANT
Ownership (Public, Private or Both Public and Private)	105b	☐ PUB ☒ PRV ☐ BPP
Check block if Federal Facility and give GSA Inventory Control Number	105c	☐ FED
	105d	N.A
Location		
Street & Number	105e	Rifle Range Road
City	105f	Vicksburg Ms.
County	105g	Warren
State	105h	Mississippi

6. Nature of Business State the nature of the business conducted at the plant or operating facility.

	106a	Manufacture of Organic and Inorganic Chemicals
	106b	AGENCY USE

7. Facility Intake Water (see instructions) Indicate water intake volume per day by sources. Estimate average volume per day in thousand gallons per day.

Municipal or private water system	107a	1500 thousand gallons per day
Surface water	107b	0 thousand gallons per day
Groundwater	107c	0 thousand gallons per day
Other*	107d	0 thousand gallons per day
Total Item 7	107e	1500 thousand gallons per day
*If there is intake water from 'other,' specify the source.	107f	None

8. Facility Water Use Estimate average volume per day in thousand gallons per day for the following types of water usage at the facility. (see instructions)

Noncontact cooling water	108a	840 thousand gallons per day
Boiler feed water	108b	200 thousand gallons per day
Process water (including contact cooling water)	108c	430 thousand gallons per day
Sanitary water	108d	30 thousand gallons per day
Other*	108e	- thousand gallons per day
Total Item 8	108f	1500 thousand gallons per day
*If there are discharges to 'other,' specify.	108g	NONE
If there is 'Sanitary' water use, give the number of people served.	108h	300 people served

EPA Form 7550-23 (7-73)

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9. All Facility Discharges and other Losses; Number and Discharge (see instructions) Volume Specify the number of discharge points and the volume of water discharged or lost from the facility according to the categories below. Estimate average volume per day in thousand gallons per day.

		Number of Discharge Points		Total Volume Used or Discharged, Thousand Gal/Day
Surface Water	109a1	1	109a2	450
Sanitary wastewater transport system	109b1	1	109b2	10
Storm water transport system	109c1		109c2	
Combined sanitary and storm water transport system	109d1		109d2	
Surface impoundment with no effluent	109e1		109e2	
Underground percolation	109f1		109f2	
Well injection	109g1		109g2	
Waste acceptance firm	109h1		109h2	
Evaporation	109i1	4	109i2	840
Consumption	109j1	2	109j2	200
Other*	109k1		109k2	
Facility discharges and volume Total Item 9.	109l1		109l2	1500
*If there are discharges to 'other,' specify.	109m1			

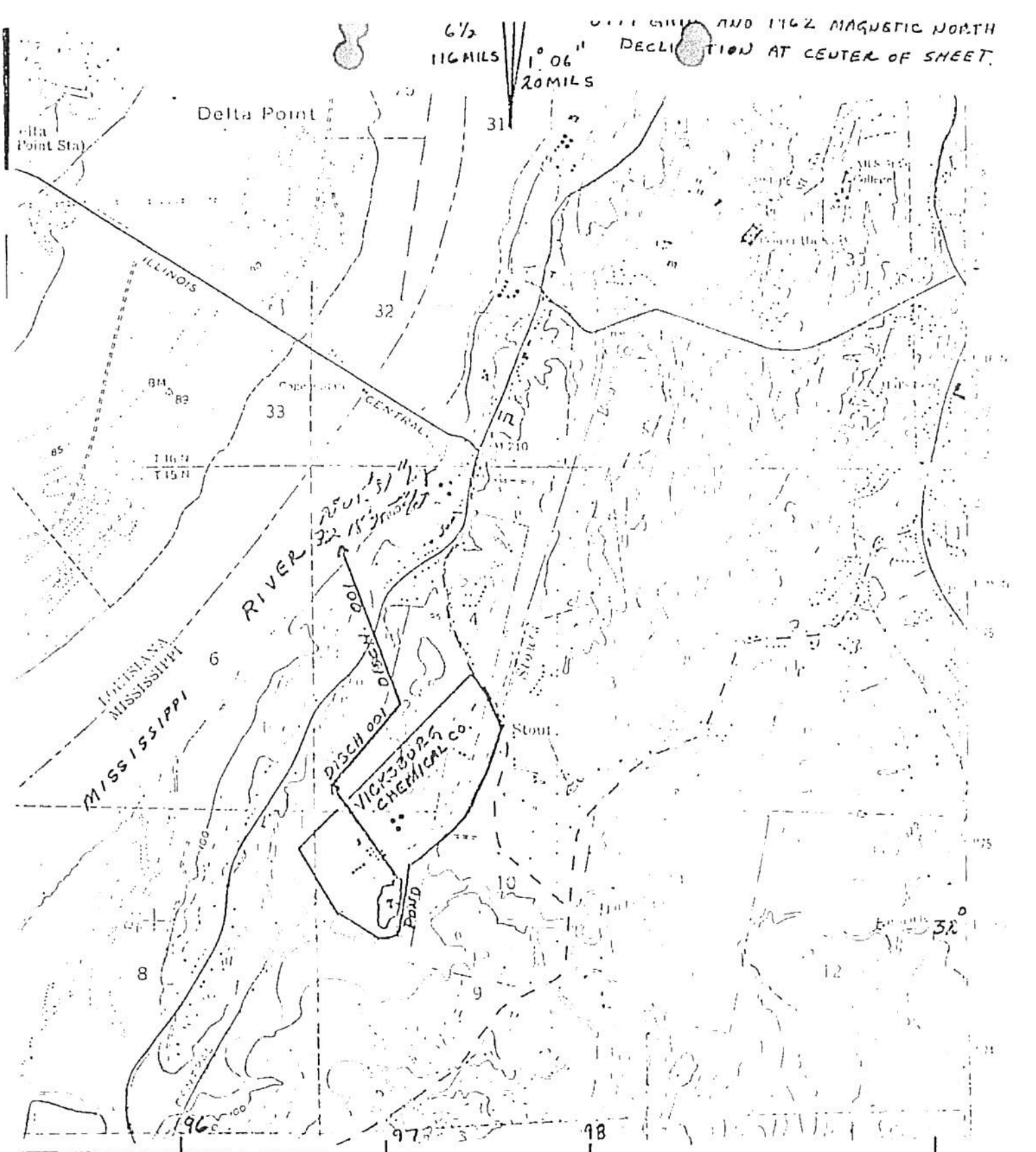
10. Permits, Licenses and Applications
List all existing, pending or denied permits, licenses and applications related to discharges from this facility (see instructions).

110	Issuing Agency (a)	For Agency Use (b)	Type of Permit or License (c)	ID Number (d)	Date Filed YR/MO/DA (e)	Date Issued YR/MO/DA (f)	Date Denied YR/MO/DA (g)	Expiration Date YR/MO/DA (h)
1.	None							
2.								
3.								

11. Maps and Drawings
Attach all required maps and drawings to the back of this application. (see instructions)

12. Additional Information

112 Item Number	Information
	None

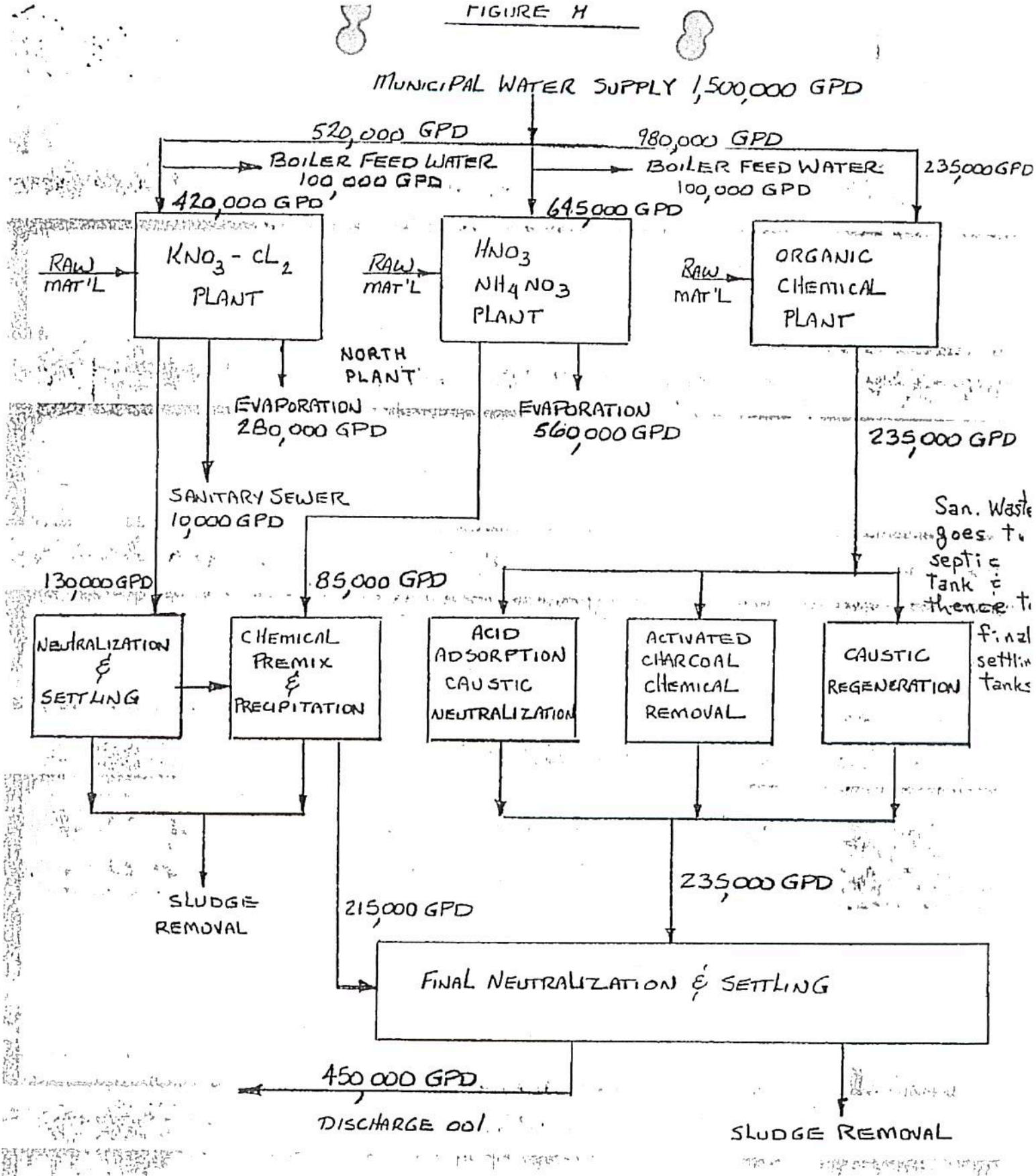


55' FIGURE B

SCALE 1: 24000

"LOCATION MAP"
FROM U.S. GEOLOGICAL SURVEY
MAP. 1972.
VICKSBURG CHEMICAL CO.
VICKSBURG, WARREN, MISS.
NOV. 13, 1973

FIGURE H



SCHEMATIC OF WATER FLOW
VICKSBURG CHEMICAL CO.
VICKSBURG, WARREN, MISS.
4-22-74 PAGE 1 OF 1

VICKSBURG CHEMICAL COMPANY

P. O. BOX 3
VICKSBURG, MISSISSIPPI 39180
PHONE (601) 636-1231

July 31, 1974

Mr. Danny Averett
State of Mississippi
Air & Water Pollution Control Commission
P.O. Box 827
Jackson, Ms. 39205

Dear Mr. Averett:

Attached please find three copies of a Block Flow Sheet illustrating the effluent quantities and quality which will be discharged from the Unsymetrical Dimethyl Hydrazine(UDMH) Modifications currently in progress at the existing Methyl Parathion complex.

As discussed with you on 7-18-74, this product will be manufactured for the U.S. Government. UDMH is a primary fuel component for liquid fired missile, i.e., the "Titan", the surface-to-surface "Lance" missile system. Lance is a NATO weapon utilized by all NATO countries. Other missile systems which utilize UDMH are vital to National Defense. At present, the government supply is critically low, and no other company has either the equipment or technology to supply this need on a rapid basis. The urgency of this requirement is relatively short lived, i.e., approximately six months. Vicksburg Chemical has been asked to supply this requirement during this period.

This will require Vicksburg Chemical to go into manufacturing operations immediately. At the end of the six month period, UDMH manufacture at Vicksburg will cease.

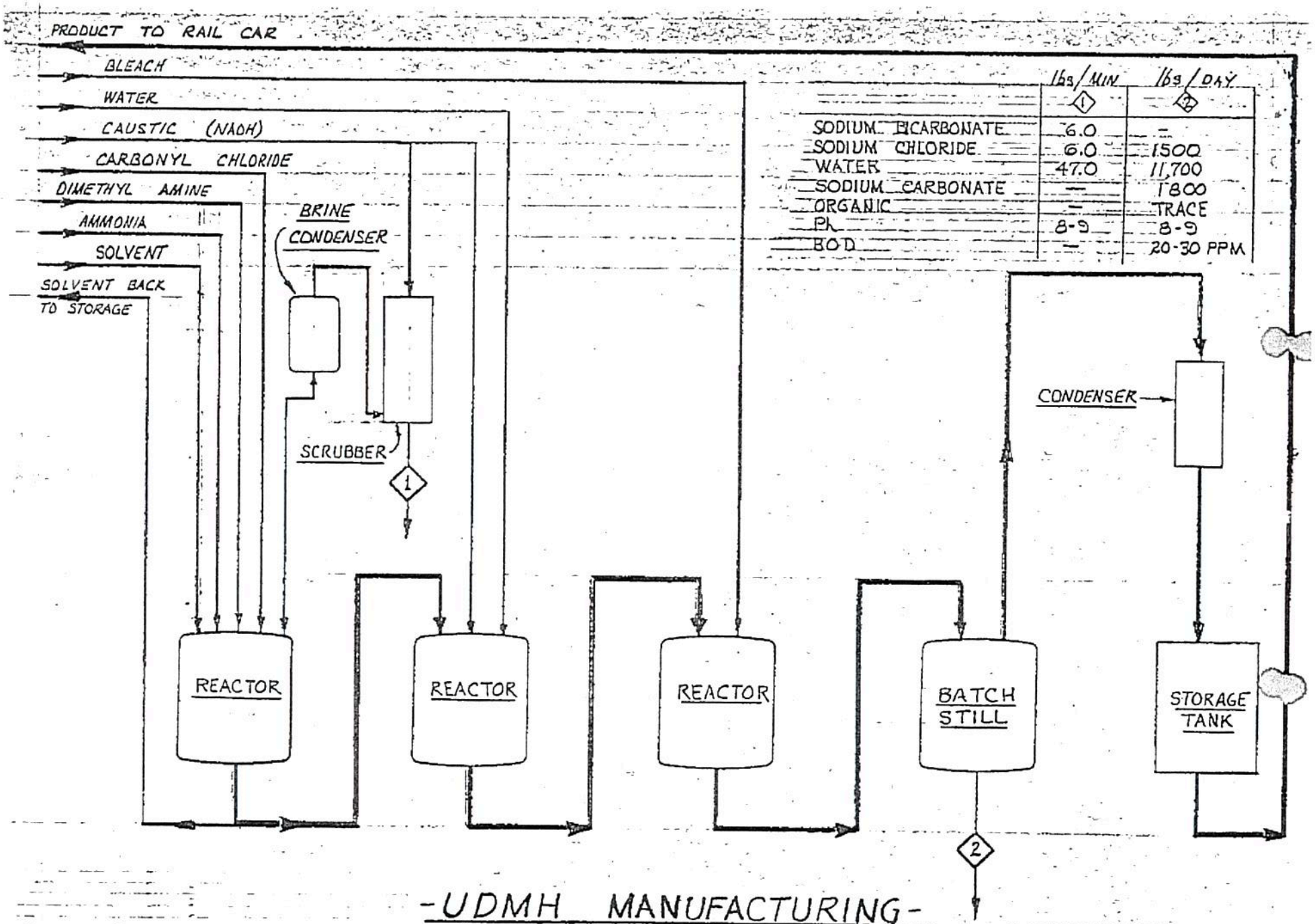
Based upon our observations, effluent discharges from UDMH will not significantly add to current effluent discharges from the existing Parathion complex. Due to the urgency of this product requirement, immediate approval is requested.

Sincerely,

VICKSBURG CHEMICAL CO.

James A. Alexander
Engineering Manager

JAA:dc



	lbs/MIN ①	lbs/DAY ②
SODIUM BICARBONATE	6.0	–
SODIUM CHLORIDE	6.0	1500
WATER	47.0	11,700
SODIUM CARBONATE	–	1800
ORGANIC	–	TRACE
Ph	8-9	8-9
BOD	–	20-30 PPM

-UDMH MANUFACTURING-

MODIFICATION TO EXISTING METHYL PARATHION PLANT

Vicksburg Chemical Co.
VICKSBURG, MISSISSIPPI 39180

FORM APPROVED
OMB No. 158-R0100

STANDARD FORM C – MANUFACTURING AND COMMERCIAL

FOR AGENCY USE
MS0027995

SECTION II. BASIC DISCHARGE DESCRIPTION

Complete this section for each discharge indicated in Section I, Item 9, that is to surface waters. This includes discharges to municipal sewerage systems in which the wastewater does not go through a treatment works prior to being discharged to surface waters. Discharges to wells must be described where there are also discharges to surface waters from this facility. **SEPARATE DESCRIPTIONS OF EACH DISCHARGE ARE REQUIRED EVEN IF SEVERAL DISCHARGES ORIGINATE IN THE SAME FACILITY.** All values for an existing discharge should be representative of the twelve previous months of operation. If this is a proposed discharge, values should reflect best engineering estimates.

ADDITIONAL INSTRUCTIONS FOR SELECTED ITEMS APPEAR IN SEPARATE INSTRUCTION BOOKLET AS INDICATED. REFER TO BOOKLET BEFORE FILLING OUT THESE ITEMS.

1. Discharge Serial No. and Name		
a. **Discharge Serial No.** (see Instructions)	201a	001
b. **Discharge Name** Give name of discharge, if any. (see Instructions)	201b	VICKSBURG CHEMICAL CO. OUT FALL
c. **Previous Discharge Serial No.** If previous permit application was made for this discharge (see Item 4, Section I), provide previous discharge serial number.	201c	NONE
2. Discharge Operating Dates		
a. **Discharge Began Date** If the discharge described below is in operation, give the date (within best estimate) the discharge began.	202a	PRIOR TO 1953 (YR MO)
b. **Discharge to Begin Date** If the discharge has never occurred but is planned for some future date, give the date (within best estimate) the discharge will begin.	202b	N A (YR MO)
c. **Discharge to End Date** If discharge is scheduled to be discontinued within the next 5 years, give the date (within best estimate) the discharge will end.	202c	N A (YR MO)
3. Engineering Report Available Check if an engineering report is available to reviewing agency upon request. (see Instructions)	203	☐ NONE

4. Discharge Location Name the political boundaries within which the point of discharge is located.

				Agency Use
State	204a	MISSISSIPPI	204d	
County	204b	WARREN	204e	
(if applicable) City or Town	204c	VICKSBURG	204f	

5. Discharge Point Description Discharge is into (check one): (see Instructions)

Stream (includes ditches, arroyos, and other intermittent watercourses)	205a	☒ STR
Lake		☐ LKE
Ocean		☐ OCE
Municipal Sanitary Wastewater Transport System		☐ MTS
Municipal Combined Sanitary and Storm Transport System		☐ MCS

FOR AGENCY USE
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Municipal Storm Water Transport System		☐ STS
Well (Injection)		☐ WEL
Other		☐ OTH
If 'other' is checked, specify	205b	

6. **Discharge Point — Lat/Long** Give the precise location of the point of discharge to the nearest second.

Latitude	206a	32 DEG 18 MIN 25 SEC
Longitude	206b	90 DEG 05 MIN 31 SEC

7. **Discharge Receiving Water Name** Name the waterway at the point of discharge.(see instructions)

207a MISSISSIPPI RIVER

If the discharge is through an outfall that extends beyond the shoreline or is below the mean low water line, complete Item 8.

207b

For Agency Use			207c	For Agency Use
Major	Minor	Sub		303e

8. **Offshore Discharge**

a. Discharge Distance from Shore	208a	NONE feet
b. Discharge Depth Below Water Surface	208b	NONE feet

9. **Discharge Type and Occurrence**

a. Type of Discharge Check whether the discharge is continuous or intermittent. (see instructions) — 209a
☒ (con) Continuous
☐ (int) Intermittent

b. Discharge Occurrence Days per Week Enter the average number of days per week (during periods of discharge) this discharge occurs. — 209b: 7 days per week

c. Discharge Occurrence —Months If this discharge normally operates (either intermittently, or continuously) on less than a year-around basis (excluding shutdowns for routine maintenance), check the months during the year when the discharge is operating. (see instructions) — 209c

☐ JAN ☐ FEB ☐ MAR ☐ APR
☐ MAY ☐ JUN ☐ JUL ☐ AUG
☐ SEP ☐ OCT ☐ NOV ☐ DEC

Complete Items 10 and 11 if "Intermittent" is checked in Item 9.a. Otherwise, proceed to Item 12.

10. **Intermittent Discharge Quantity** State the average volume per discharge occurrence in thousands of gallons. — 210: N/A thousand gallons per discharge occurrence.

11. **Intermittent Discharge Duration and Frequency**

a. Intermittent Discharge Duration Per Day State the average number of hours per day the discharge is operating.	211a	N/A hours per day
b. Intermittent Discharge Frequency State the average number of discharge occurrences per day during days when discharging.	211b	N/A discharge occurrences per day

12. **Maximum Flow Period** Give the time period in which the maximum flow of this discharge occurs. — 212: From JAN (month) to JAN (month)

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13. Activity Description Give a narrative description of activity producing this discharge.(see instructions)

213a 1) Manufacture of KNO_3-Cl_2 by Reaction of KCL W/ HNO_3 2) Manufacture of HNO_3 by NH_3 process;(3) Manufacture of NH_4NO_3 by Reaction of HNO3 W/NH_3;(4) Manufacture Toxaphene by Reaction of camphene W/Cl_2(5) Manufacture of Dinitro Buty Phenol by Nitric Acid-Secondary Phenol Reaction(6) Manufactu of Methyl Parathion(7) Manufacture of substituted Triazenes by Reaction of cyanuric chloride W/Ethanol & 150 Propyl Amin (8) Manufactures of Cyanuric chloride by Reaction of Hydroge Cyanide W/Chlorine(9)Manufacture of Maloran by Reaction of M-Chlorophenylisocyanate W/Organic Sulfates & Bromine(10) Ma ufacture of complex organic intermediates by use of Aromatic & Aliphatic organic derivatives W/Inorganic & organic compounds.

14. Activity Causing Discharge For each SIC Code which describes the activity causing this discharge, supply the type and maximum amount of either the raw material consumed (Item 14a) or the product produced (Item 14b) in the units specified in Table I of the Instruction Booklet. For SIC Codes not listed in Table I, use raw material or production units normally used for measuring production.(see instructions)

a. Raw Materials

	SIC Code	Name	Maximum Amount/Day	Unit (See Table I)	Shared Discharges (Serial Number)
214a	(1)	(2)	(3)	(4)	(5)
	N/A				

b. Products

	SIC Code	Name	Maximum Amount/Day	Unit (See Table I)	Shared Discharges (Serial Number)
214b	(1)	(2)	(3)	(4)	(5)
	2812	Potassium Nitrate	300	K-1	001
	2812	Chlorine	100	K-1	001
	2812	Nitric Acid	200	K-1	001
	2873	Ammonium Nitrate	20	O-1	001
	2879	Toxaphene	35	R-1	001
	2879	DNBP	25	R-1	001
	2879	Methyl Parathion	20	R-1	001
	2879	Triazenes	70	R-1	001
	2879	Maloran	9	R-1	001
	*2765	UDMH	4	P-1	001

FOR AGENCY USE

m	s	0	0	2	7	9	9	s

15. Waste Abatement

a. Waste Abatement Practices
Describe the waste abatement practices used on this discharge with a brief narrative. (see instructions)

215a

Narrative: Acid, chlorine & nitrogen Tetroxide are scrubbed with sodium Hydroxide, Phenols are adsorbed in activated carbon which is dischargedto a commercial effluent company; organo phosphates are reacted with sodium hydroxide; sodium hypochlorite & Ammonia are premixed to eliminate free chlorine ammonia & the mixed effluents go to a final neutralization settling Pond prior to discharge

b. Waste Abatement Codes
Using the codes listed in Table II of the Instruction Booklet, describe the waste abatement processes for this discharge in the order in which they occur if possible.

215b

(1) CNEUTR,	(2) NADSOR,	(3) CMTDRO,
(4) CCLDIS,	(5) PSEDIM,	(6) ____,
(7) ____,	(8) ____,	(9) ____,
(10) ____,	(11) ____,	(12) ____,
(13) ____,	(14) ____,	(15) ____,
(16) ____,	(17) ____,	(18) ____,
(19) ____,	(20) ____,	(21) ____,
(22) ____,	(23) ____,	(24) ____,
(25) ____.		

*Unsymetrical Dimethyl Hydrazine(UDMH) production is limited to 6 months production. At the end of this time UDMH production will cease at Vicksburg. Effects of UDMH effluent on total plant effluent during this interim production are attached in correspondence form, dated 7-31-74, & 8-12-74.

DISCHARGE SERIAL NUMBER

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16. Wastewater Characteristics

Check the box beside each constituent which is present in the effluent (discharge water). This determination is to be based on actual analysis or best estimate.(see instructions)

Parameter 216	Present	Parameter 216	Present
Color 00080	X	Copper 01042	
Ammonia 00610	X	Iron 01045	X
Organic nitrogen 00605	X	Lead 01051	
Nitrate 00620	X	Magnesium 00927	
Nitrite 00615		Manganese 01055	
Phosphorus 00665		Mercury 71900	
Sulfate 00945	X	Molybdenum 01062	
Sulfide 00745	X	Nickel 01067	
Sulfite 00740		Selenium 01147	
Bromide 71870	X	Silver 01077	
Chloride 00940	X	Potassium 00937	X
Cyanide 00720		Sodium 00929	X
Fluoride 00951		Thallium 01059	
Aluminum 01105		Titanium 01152	
Antimony 01097		Tin 01102	
Arsenic 01002		Zinc 01092	
Beryllium 01012		Algicides* 74051	X
Barium 01007		Chlorinated organic compounds* 74052	
Boron 01022		Pesticides* 74053	X
Cadmium 01027		Oil and grease 00550	
Calcium 00916		Phenols 32730	X
Cobalt 01037		Surfactants 38260	
Chromium 01034		Chlorine 50060	X
Fecal coliform bacteria 74055		Radioactivity* 74050	

*Specify substances, compounds and/or elements in Item 26.

Pesticides (insecticides, fungicides, and rodenticides) must be reported in terms of the acceptable common names specified in *Acceptable Common Names and Chemical Names for the Ingredient Statement on Pesticide Labels*, 2nd Edition, Environmental Protection Agency, Washington, D.C. 20250, June 1972, as required by Subsection 162.7(b) of the Regulations for the Enforcement of the Federal Insecticide, Fungicide, and Rodenticide Act.

DISCHARGE SERIAL NUMBER

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17. Description of Intake and Discharge

For each of the parameters listed below, enter in the appropriate box the value or code letter answer called for.(see instructions)

In addition, enter the parameter name and code and all required values for any of the following parameters if they were checked in Item 16; ammonia, cyanide, aluminum, arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, zinc, phenols, oil and grease, and chlorine (residual).

Parameter and Code 217a	Influent		Effluent					
	Untreated Intake Water (Daily Average) (1)	In-Plant Treated Intake Water (Daily Average) (2)	Daily Average (3)	Minimum Value Observed or Expected During Discharge Activity (4)	Maximum Value Observed or Expected During Discharge Activity (5)	Frequency of Analysis (6)	Number of Analyses (7)	Sample Type (8)
Flow* Gallons per day 00056	1500	200						
pH Units 00400	8.4	7.6	X	6	10	5/7	260	G
Temperature (winter) ° F 74028	65		78	75	85			
Temperature (summer) ° F 74027	65		88	83	93			
Biochemical Oxygen Demand (BOD 5-day) mg/l 00310			50	30	150	1/7	2	G
Chemical Oxygen Demand (COD) mg/l 00340			450	300	700	1/7	6	G
Total Suspended (nonfilterable) Solids mg/l 00530			70	25	300			
Specific Conductance micromhos/cm at 25° C 00095			X					
Settleable Matter (residue) ml/l 00545								

*Other discharges sharing intake flow (serial numbers).(see instructions)

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MS 00279 95

17. (Cont'd.)

Parameter and Code 217a	Influent		Effluent					
	Untreated Intake Water (Daily Average) (1)	In-Plant Treated Intake Water (Daily Average) (2)	Daily Average (3)	Minimum Value Observed or Expected During Discharge Activity (4)	Maximum Value Observed or Expected During Discharge Activity (5)	Frequency of Analysis (6)	Number of Analyses (7)	Sample Type (8)
Ammonia as N 00610			150	0	300	1/7	52	G
Phenols 32730			0.1	0	15	3/7	150	G
Oil & Greases 00550			0.1	0.1	0.3	1/7	50	G
Chlorine 50060			6.0	0.1	10	0.5/7	26	G
Nitrate as N 00625			750	150	1500	1/7	52	G

18. **Plant Controls** Check if the following plant controls are available for this discharge. 218

Alternate power source for major pumping facility. ☐ APS

Alarm or emergency procedure for power or equipment failure ☒ ALM

Complete item 19 if discharge is from cooling and/or steam water generation and water treatment additives are used.

19. **Water Treatment Additives** If the discharge is treated with any conditioner, inhibitor, or algicide, answer the following:

a. Name of Material(s) 219a J-12, C-30, 605

b. Name and address of manufacturer 219b Betz, Inc.
4636 Somerton Rd.
Trevose, Pa. 19407

c. Quantity (pounds added per million gallons of water treated). 219c

DISCHARGE SERIAL NUMBER

FOR AGENCY USE
MS0027995

d. Chemical composition of these additives (see instructions). | 219d | Quaternary Ammonium TriButyl tin, Methylene Bisbicyanate Bis Chloro Methyl Sulfone & Hexlethyl Di Phosphonate, Magneisium Oxide, Sodium Carbonate

Complete items 20-25 if there is a thermal discharge (e.g., associated with a steam and/or power generation plant, steel mill, petroleum refinery, or any other manufacturing process) and the total discharge flow is 10 million gallons per day or more. (see instructions)

20. Thermal Discharge Source Check the appropriate item(s) indicating the source of the discharge. (see instructions) | 220 | N/A

- Boiler Blowdown ☐ BLBD
- Boiler Chemical Cleaning ☐ BCCL
- Ash Pond Overflow ☐ APOF
- Boiler Water Treatment — Evaporator Blowdown ☐ EPBD
- Oil or Coal Fired Plants - Effluent from Air Pollution Control Devices ☐ OCFP
- Condense Cooling Water ☐ COND
- Cooling Tower Blowdown ☐ CTBD
- Manufacturing Process ☐ MFPR
- Other ☐ OTHR

21. Discharge/Receiving Water Temperature Difference

Give the maximum temperature difference between the discharge and receiving waters for summer and winter operating conditions. (see instructions)

- Summer | 221a | 32 °F.
- Winter | 221b | 22 °F.

22. Discharge Temperature, Rate of Change Per Hour | 222 | N/A °F./hour

Give the maximum possible rate of temperature change per hour of discharge under operating conditions. (see instructions)

23. Water Temperature, Percentile Report (Frequency of Occurrence) N/A

In the table below, enter the temperature which is exceeded 10% of the year, 5% of the year, 1% of the year and not at all (maximum yearly temperature). (see instructions)

Frequency of occurrence		10%	5%	1%	Maximum
a. Intake Water Temperature (Subject to natural changes)	223a	°F	°F	°F	°F
b. Discharge Water Temperature	223b	°F	°F	°F	°F

24. Water Intake Velocity (see instructions) | 224 | N/A feet/sec.

25. Retention Time Give the length of time, in minutes, from start of water temperature rise to discharge of cooling water. (see instructions) | 225 | N/A minutes

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FOR AGENCY USE

MS	0	0	0	7	9	9	5

26. Additional Information

226

Item	Information
1	All intake water is tap water from the city of Vicksburg Mississippi.
2	In-Plant treated water is the above tap water treated to remove SiO_2 & hardness for boiler feed water.
3	Pesticides Referenced in item 16 are:
	Dinitro Butyl Phenol (herbicide)
	Triazines (herbicide)
	Methyl Parathion (insecticide)
	Maloran (herbicide)

VICKSBURG CHEMICAL COMPANY

P. O. BOX 3
VICKSBURG, MISSISSIPPI 39180
PHONE (601) 636-1231

August 12, 1974

Mr. Danny Averett
State of Mississippi
Air & Water Pollution Control Commission
P.O. Box 827
Jackson, Ms. 39205

Dear Mr. Averett:

Per your telephone request of August 1, 1974, the following supplemental data is supplied concerning the manufacture of UDMH in the existing Methyl Parathion complex. Please note that the total solids increase is due to sodium chloride, sodium carbonate, & sodium bicarbonate shown on the sketch supplied to your office on July 31, 1974. Hydrazine reports are decomposition products of UDMH, and are mainly Dimethyl Amines with trace amouts of hydrazine.

As pointed out of August 1, 1974, the duration of this project is six months. At the end of this period, UDMH manufacture will cease and the unit will be restored to its normal purpose. Should there be any further question, do not hesitate to contact me.

Sincerely,

VICKSBURG CHEMICAL CO.

JAA:dc

James A. Alexander
Engineer Manager

VICKSBURG CHEMICAL

VICKSBURG, MS.

UDMH MANUFACTURE

EXISTING METHYL PARATHION MANUFAXTURING FACILITIES

SUPPLEMENTAL EFFLUENT DATA

Effluent from UDMH Manufacture mg/l		Total Plant Effluent mg/l Prior to UDMH	With UDMH	Increase
Ammonia-None	Total Solids	6,493	11,696	5,203 *
Organic Nitrogen 488 ppm	Chlorides	2,360	3,148	788
Free Chlorine-None	BOD_5	50	55	5
COD-1,952	NH_3 Nitrogen	150	0	0
Oil/Greases-None	Hydrazines **	-	12.1	12.1
	Organic Nitrogen	-	6.1	6.1

*Increase in solids due to sodium chloride, sodium carbonate & sodium bicarbonate

**Hydrazine listed is decomposition Products of Unsymetrical Dimethyl Hydrazine; major constituant is Dimethyl Amine.

Enclosure 4

Georgia Department of Natural Resources
205 Butler Street, S.E., Floyd Towers East, Atlanta, Georgia 30334
J. Leonard Ledbetter, Commissioner
Harold F. Reheis, Assistant Director
Environmental Protection Division

REFERENCE 3

August 17, 1988

Mr. Glenn R. Hoffmann
Hercules Incorporated
P.O. Drawer 1517
Brunswick, GA 31521

Dear Mr. Hoffmann:

This letter is in response to the assessment plan for the plant wastewater equalization basin and the toxaphene clay-silica sludge surface impoundments submitted to the Division on June 16, 1988.

Representatives of the Georgia Environmental Protection Division (EPD) took samples from the equalization basin and drainage ditch on May 13, 1988. This sampling showed high levels of several constituents in the equalization basin and from the overflow drainage ditch. The following compounds were detected;

	Equalization Basin		Drainage Ditch	
Constituent	Sludge	Water	Sludge	Water
Toxaphene	80.4mg/kg	BDL	10.4 mg/kg	BDL
Carbon Tet	BDL	11 ug/l		BDL
Ethylbenzene	2 mg/kg	1500 ug/l	143 mg/kg	22 ug/l
xylene (total)	3 mg/kg	3000 ug/l	600 mg/kg	15 ug/l
p-Isopropyl Toluene	BDL	2500 ug/l	16,000 mg/kg	4 ug/l
Benzene	BDL	48 ug/l	BDL	3 ug/l
Chlorobenzene	BDL	BDL	BDL	1 ug/l
Acetone	BDL	BDL	BDL	51 ug/l
Toluene	BDL	1300 ug/l	BDL	BDL
MIBK	BDL	32,7000 ug/l	BDL	BDL
Isopropyl Benzene	BDL	624 ug/l	BDL	BDL
Barium (total)	20 mg/kg	35 mg/l	48 mg/kg	700 mg/l

Also, both water and sludge from the drainage ditch and the equalization basin contained numerous compounds with spectra similiar to turpentine components.

Hercules should submit the results of the OCPSF priority pollutant scan of the wastewater as soon as the results are received.

To more accurately determine the groundwater quality at the equalization basin the Division recommends the installation of three cluster wells monitoring both the top of the lower confining unit of the upper most aquifer

and the water table zone. Screen lengths on these wells should not exceed ten (10) feet. The location of these wells should be adjacent to the downgradient side of the drainage ditch that runs parallel (north-south) on the eastern side of the equalization basin. The three wells should be placed at equal distances along the entire length of the basin. As proposed in the assessment plan these wells and the upgradient well will be sampled initially for the entire priority pollutant scan and any other constituents already found in the previous samples.

The surface water and sediment sampling of the north-south ditch along the eastern boundary of the equalization basin is approved.

The assessment plan for the toxaphene sludge surface impoundment has been reviewed. All wells that show toxaphene levels below the 5.0 ppb drinking water standard must be sampled semi annually for a period of three years. As outlined in the assessment plan, should an increase in toxaphene or chlorides in T-1D and T-1S or an increase in toxaphene in wells T-2 through T-6 occur above the drinking water standards, additional action may be required.

Please submit the updated assessment plan for the plant wastewater equalization basin within 45 days of receipt of this letter. Should you have any questions about the above requirements please contact Jon Olivier at 656-2833.

Sincerely,

Jim Ussery
Unit Coordinator
Hazardous Waste Management Program

JU:jom:1800T

cc: Behrooz Khaleghi
Jon Olivier

File: Hercules Brunswick, Georgia